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M-58-65-2

DESIGN AND FABRICATION OF A TRACE CONTAMINANT REMOVAL SYSTEM FOR APOLLO

PHASE II REPORT PREPARED FOR
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

CONTRACT NAS 9-3415

BIOASTRONAUTICS

Lockheed

MISSILES & SPACE COMPANY

A GROUP DIVISION OF LOCKHEED AIRCRAFT CORPORATION

SUNNYVALE, CALIFORNIA

FOREWORD

This report describes the work accomplished between March and September 1965 by the Lockheed Missiles & Space Company (LMSC) under Contract NAS 9-3415, "Design and Fabrication of a Trace Contaminant Removal System for Apollo".

The work reported herein constitutes Phase II of a three phase program being conducted for the Crew Systems Division of the NASA Manned Spacecraft Center.

The Phase I effort encompassed theoretical and experimental efforts in sorption and catalysis; engineering analysis, optimization, and prototype hardware design; and test planning for final evaluation of the hardware.

The Phase II effort, reported herein, encompassed the fabrication, assembly, and experimental evaluation of the prototype hardware. The total program has been directed at LMSC by J. M. Smith. The Phase I experimental work was directed by Dr. E. V. Ballou. The Phase II fabrication, assembly and evaluation effort was directed by T. M. Olcott. Mr. Murline Owen of the Crew Systems Division was project monitor for the Manned Spacecraft Center.

Phase III will encompass (1) the acquisition of additional data to verify the "potential-plot" correlation for the adsorption capacity of several materials, (2) a study of the possible correlation of contaminant retention-time with both saturation capacity and dynamic contaminant removal behavior, and (3) additional testing of contaminant oxidation catalysts.

ABSTRACT

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The hardware designed during Phase I was constructed and evaluated. The evaluation was done in three steps: (1) a bench test of the main sorbent canister alone, (2) a bench test of the catalytic oxidizer – post sorbent assembly, and (3) the closed-chamber testing of the complete system under simulated Apollo cabin atmosphere conditions in a 200 ft^3 volume.

In this report the hardware is described, together with the test procedures, apparatus, and results. The installation of the contaminant removal system in the Apollo Command Module is described, together with the additional hardware development required prior to entering the flight qualification phase.

Author

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Section 1
INTRODUCTION AND SUMMARY

1.1 INTRODUCTION

In the event that the build-up of atmospheric trace contaminants in the Apollo during a two-week flight becomes a hazard, it will be necessary to provide a system to remove and/or control at acceptable levels the contaminants produced during these flights. The limited data on sources of contaminants, identity, generation rates, and threshold values as related to spacecraft atmospheres somewhat impede the development of such a system; but since an untenable delay would result if this development effort was postponed until extensive data are accumulated, it was deemed necessary by NASA to initiate a program for system development based largely on information presently available. The objective of this program is to synthesize, design, fabricate, and test a functional prototype contaminant control system capable of being integrated with the Apollo environmental control system.

The program has been divided into three phases. Phase I, reported in Ref. 1, covered (1) the classification of contaminants according to method of removal, (2) the selection and screening of candidate sorbents and catalysts, (3) the acquisition of experimental data to support the system analysis and design, (4) the analysis, optimization, and design of the prototype contaminant control system, and (5) the planning of an experimental program to evaluate the prototype hardware.

Phase II, reported herein, included fabrication of the hardware, evaluation of system components separately and in combination, and further consideration of hardware installation in the Apollo Command Module and integration with the Apollo environmental control unit.

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1. "Design and Fabrication of a Trace Contaminant Removal System for Apollo," M-58-65-1, Lockheed Missiles & Space Co., Sunnyvale, Calif., March 15, 1965

Phase III, presently underway, is concerned with additional verification of Polanyi "potential plot" approach developed during Phase I, an attempt at correlation of retention-time data with the results of flow-type experiments, and additional evaluation of particular oxidation catalysts under specific conditions.

1.2 SUMMARY

During Phase II, the equipment designed during Phase I was constructed and subjected to a series of tests to determine its performance characteristics. The hardware constructed is briefly described herein. The test objectives, procedure, apparatus and results are described in detail. The installation of the contaminant removal system in the Apollo Command Module is described, together with the additional hardware development required prior to entering the flight qualification phase.

Section 2
HARDWARE DESCRIPTION

The equipment described in Section 8 of Ref. 1 was fabricated during Phase II, primarily within the LMSC manufacturing organization. The major purchased item was the regenerative heat exchanger, supplier part number A 33D-92. This unit was purchased from the Janitrol Aero Div. of the Midland-Ross Corp. and is described in Ref. 2. Performance test and analysis data are contained in Ref. 3. A photograph of this unit appears as Figure 2-1.

The assembly comprising the main and post sorbent canisters, catalytic oxidizer, flow shutoff valve, and flow transducer is shown in front and rear views as Figures 2-2 and 2-3.

2.1 MAIN SORBENT CANISTER

As described in Section 8 of Ref. 1, the main sorbent canister contains a fan which produces a flow of 10 cfm through the loaded canister. The fan requires 7.5 watts and operates from a 115 V, 400 cps, single phase electrical source. The design charcoal load is 8 lb of 4 × 10 mesh Barnebey Cheney BD, impregnated with phosphoric acid per Ref. 4. A Dacron filter and spring-loaded backup plates are provided to prevent (1) charcoal dust from entering the cabin atmosphere and (2) settling of the charcoal load due to handling and acceleration loads.

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2. " Specification for Catalytic Burner Heat Exchanger", R-66723, Lockheed Missiles & Space Co., Sunnyvale, Calif., 15 Feb. 1965
3. "Test Report For Performance Test and Design Analysis of Janitrol Aero Division A 33D92 Heat Exchanger Assembly", Report No. 676, Janitrol Aero Div., Midland-Ross Corp., Columbus, Ohio, June 1965
4. "Procedure for Impregnation of Activated Charcoal", R-66888, Lockheed Missiles & Space Co., Sunnyvale, Calif., 1 March 1965

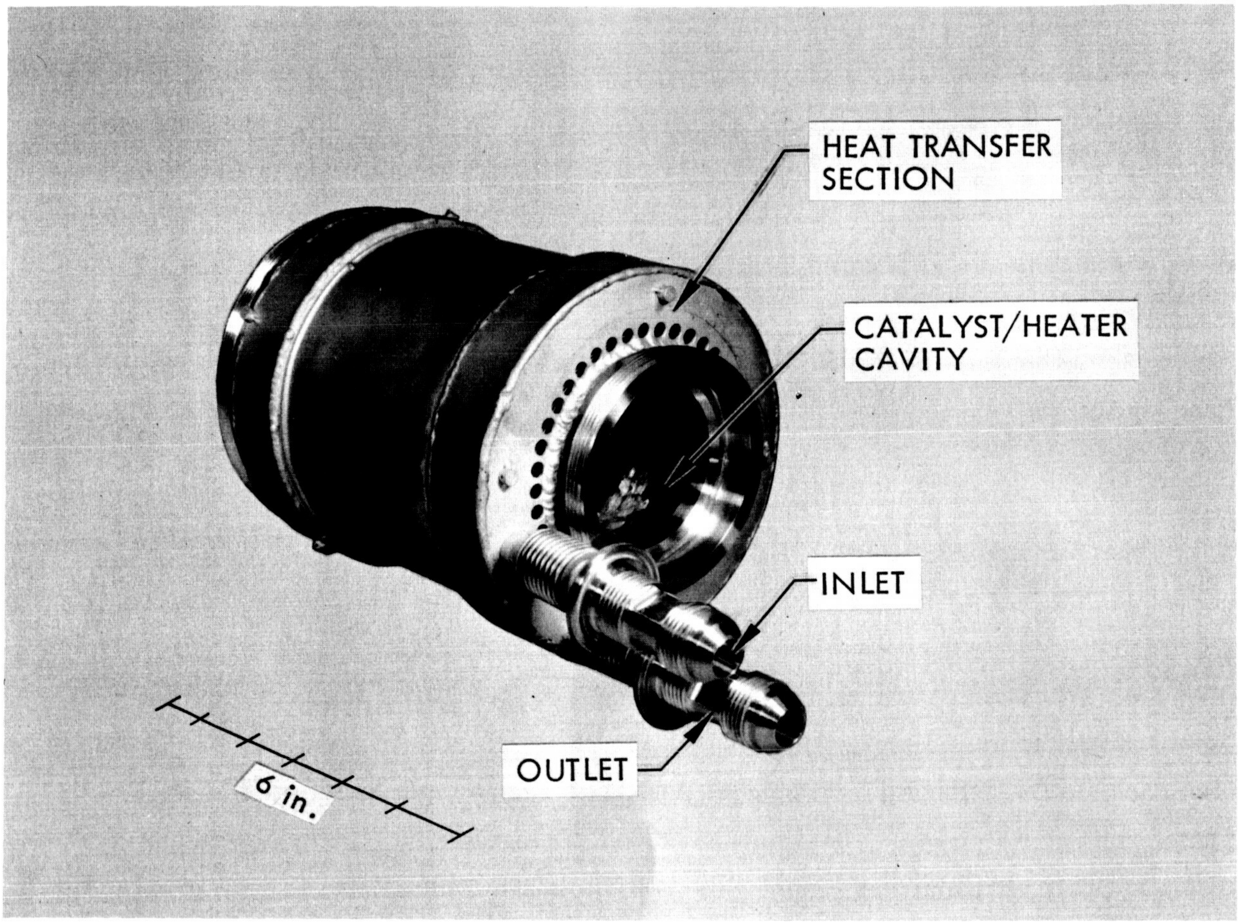

Figure 2-1 Catalytic Oxidizer Regenerative Heat Exchanger

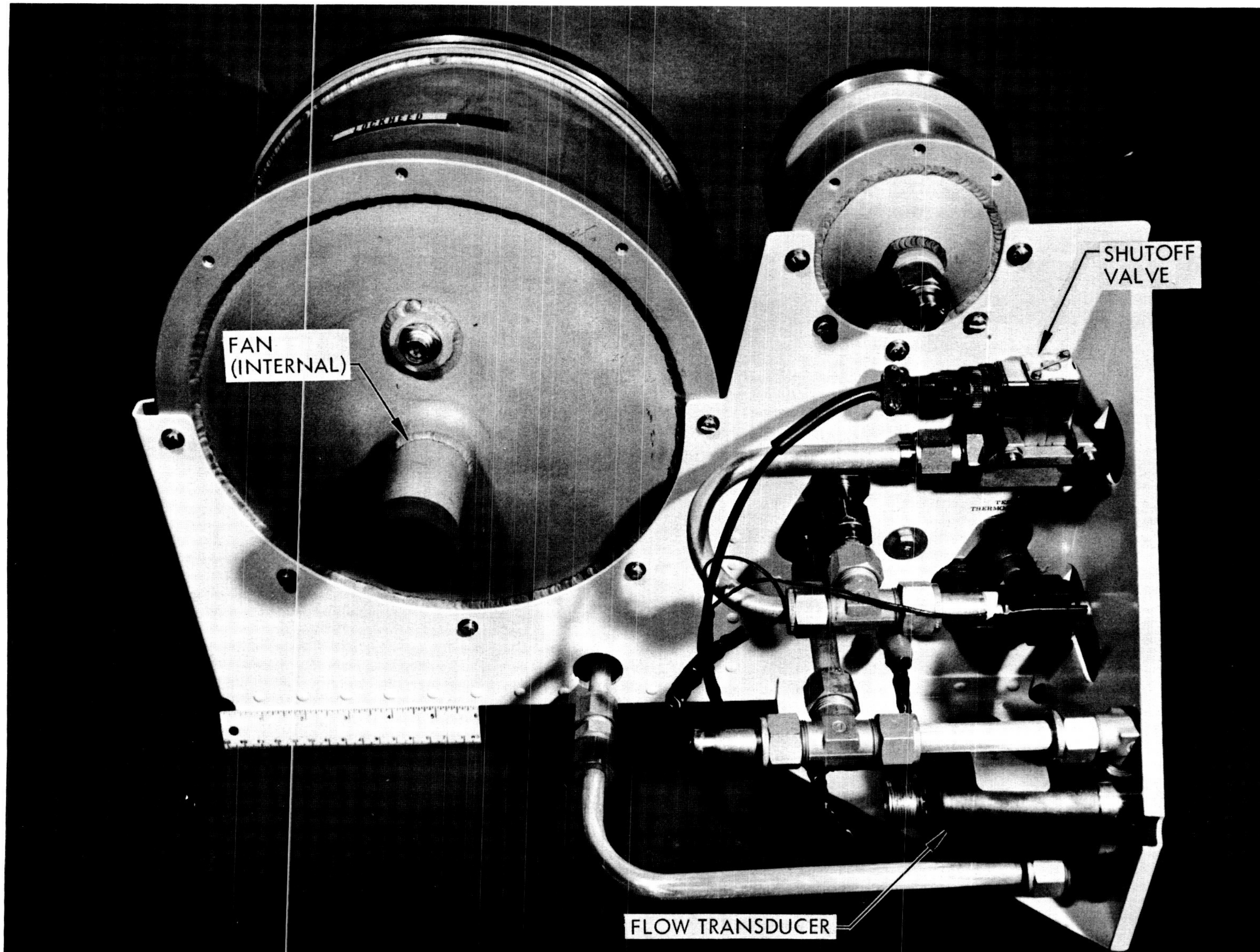

Figure 2-2 Contaminant Removal Unit – Front View

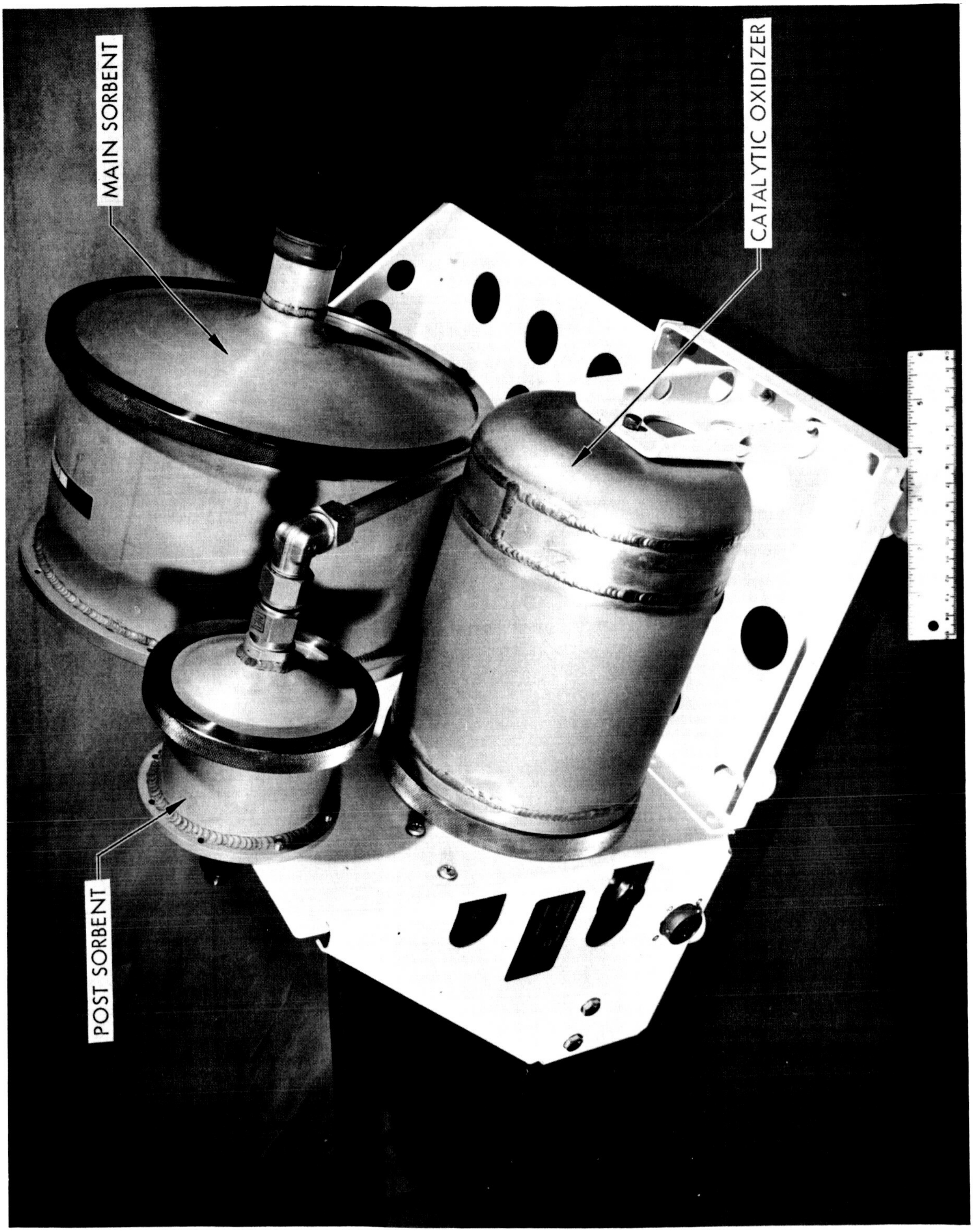

Figure 2-3 Contaminant Removal Unit – Rear View

2.2 POST SORBENT CANISTER

The post-sorbent canister contains 0.37 lb of 6 × 8 mesh LiOH. The LiOH charge is held in place by spring loaded backup plates to prevent settling. A Dacron filter is placed on the outflow side of the LiOH charge to prevent dust from entering the cabin atmosphere.

2.3 CATALYTIC OXIDIZER

The catalytic oxidizer contains 0.58 lb of 1.0% Pt. – 1.0% Pd. catalyst on 1/8" diameter Al_2O_3 spheres. Inlet flow is 1.2 cfm at 5 psia and 130°F. Pressure drop of the catalytic oxidizer/post sorbent combination is 5.5" H_2O at this flow rate. Catalyst temperature is adjustable; it is maintained at 785°F for 25% CH_4 conversion. Heating is accomplished electrically; the 28 V DC heater requires 90 watts average power to achieve a 785°F catalyst operating temperature. The regenerative heat exchanger, having an effectiveness of 83%, minimizes the gas flow heat load. Evacuated, heat-felted, fiberglass and thermal isolation mounts are provided to minimize heat leaks through the case to ambient.

2.4 FLOW SHUT-OFF VALVE

A solenoid operated valve is provided to shut off the flow of atmosphere through the catlytic oxidizer. This valve operates from a 28 V DC power source and draws 20 W when operative. Valve operation is automatically controlled by the catalyst temperature sensor.

2.5 FLOW TRANSDUCER

The flow transducer measures mass flow through the catalytic oxidizer. It is provided for test purposes only and would not be included in a flight version of the system. Flow transducer readout is displayed on the face of the controller unit.

2.6 CONTROLLER UNIT

The controller unit shown in Figure 2-4 is for test support only. Considerably fewer control and display functions would be provided for a flight version. The controller (1) allows startup and manual or automatic control of all system functions, (2) displays catalytic burner mass flow, inlet and outlet gas temperatures, and catalyst temperature, and (3) provides audio and visual alarms when an overheat condition occurs.

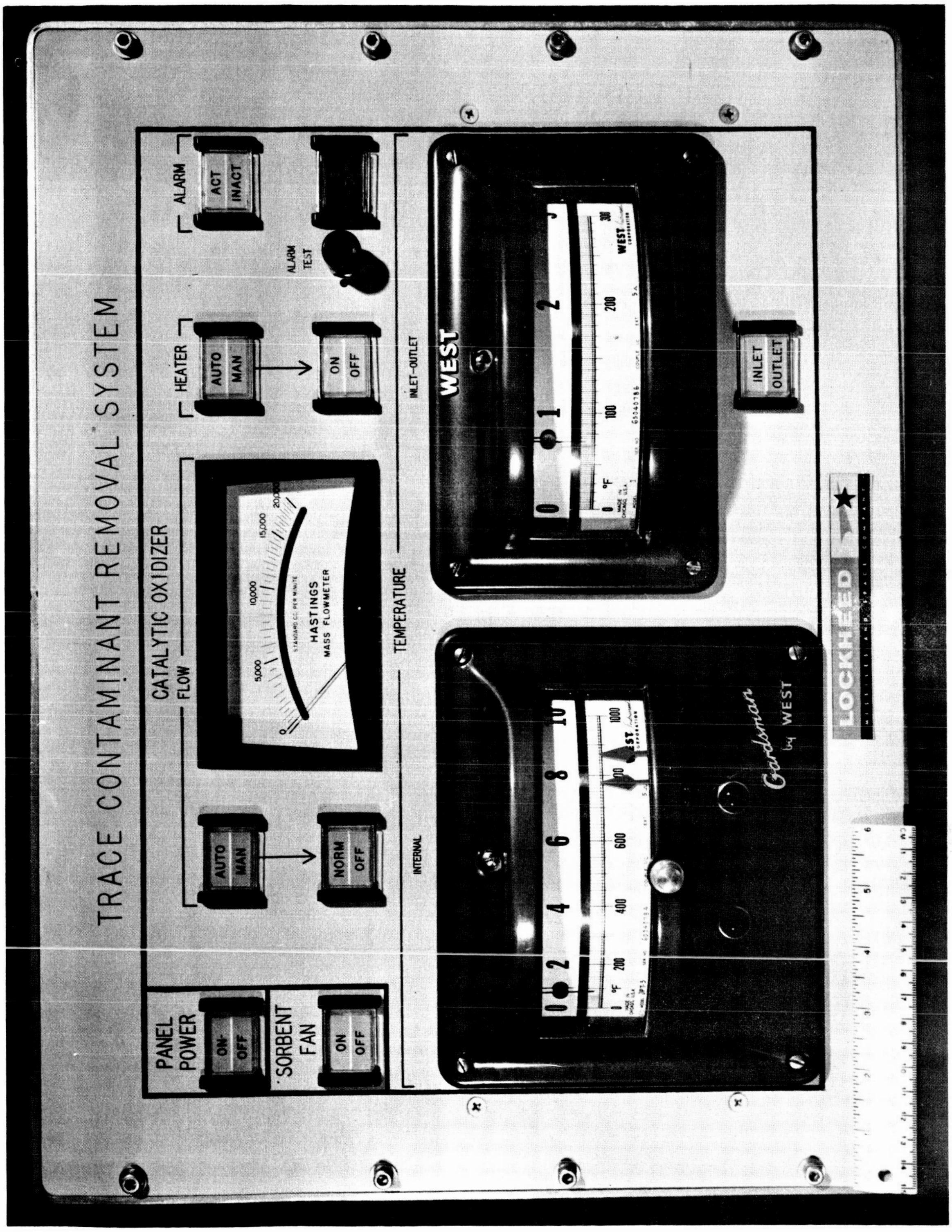

Figure 2-4 Removal System Controller for Test Support

Section 3
MAIN SORBENT TEST

3.1 OBJECTIVES

The main sorbent tests were conducted on June 8 and 9, 1965. The following section presents the objectives of the test, apparatus and procedures used, and the results obtained.

The objectives of these tests were to:

- Checkout operation of the controller
- Determine the pressure drop and fan power
- Determine the removal capability of the main sorbent for the contaminants listed in Table 3-1, under normal conditions
- Determine the removal capability of the main sorbent for n-Butane and Freon-12 under upset conditions

3.2 APPARATUS

The apparatus used in the main sorbent subsystem tests is shown in Figures 3-1 and 3-2. The equipment includes the following major elements:

- Cylinders for oxygen, gaseous contaminant, and carbon dioxide supply
- Motorized syringe for liquid contaminant introduction
- Water bubbler for humidity control
- Flow meter to measure gaseous contaminant introduction rates
- Gage to measure system total pressure
- Velocity indicator to measure system flow rate
- Atmosphere monitoring facility to perform gas analysis (described in Section 5.0)
- Draft gage to determine system pressure drop

Table 3-1

CONTAMINANTS INTRODUCED AND MEASURED – MAIN SORBENT BENCH TEST

CONTAMINANT	SMAC (mg/m^3)	ESTIMATED** INSTRUMENT SENSITIVITY (mg/m^3)	ANTICIPATED INTRODUCTION RATE (gm/day)	ACTUAL*** INTRODUCTION (gm/day)	CONTAMINANT CONCENTRATION AT 24 HR (mg/m^3)
Freon 12	130.0	0.3	0.304	0.362	2.7
1,4 Dioxane	72.0	20.0	"	0.304	*
Ammonia	7.0	1.5	1.47	1.470	≤ 1.6
Butene-1	60.4	0.3	0.304	0.362	1.2
Trans Butene-2	60.4	0.3	"	0.362	1.2
Ethyl Alcohol	50.0	10.0	"	0.304	*
n-Butyl Alcohol	60.0	37.0	"	"	*
Freon 11	147.0	0.3	"	"	*
Freon 22	93.5	0.3	0.160	0.190	5.5
Acetone	63.0	30.0	0.304	0.362	*
Ethyl Acetate	92.0	58.0	"	0.304	*
Nitrous Oxide	47.4	0.02	0.001	0.001	*
n-Hexane	94.6	10.0	0.304	0.304	*
Trichloroethylene	104.0	40.0	"	"	*
Toluene	92.0	18.0	"	"	*
Freon 21	103.0	0.3	"	0.362	*
n-Butane	26.5	0.3	"	0.362	1.2
Propyl Mercaptan	82.0	50.0	"	"	*
Methyl Chloroform	143.0	25.0	"	"	*
Phosphoric Acid	0.2	0.09	0	0	0.09
			6.500	6.989	

*None detected.
**Error approximately ±10 percent.
***The actual contaminant introduction rate in some cases was slightly different from the anticipated value due to deviations in the concentrations of purchased gas mixtures from the requested values.

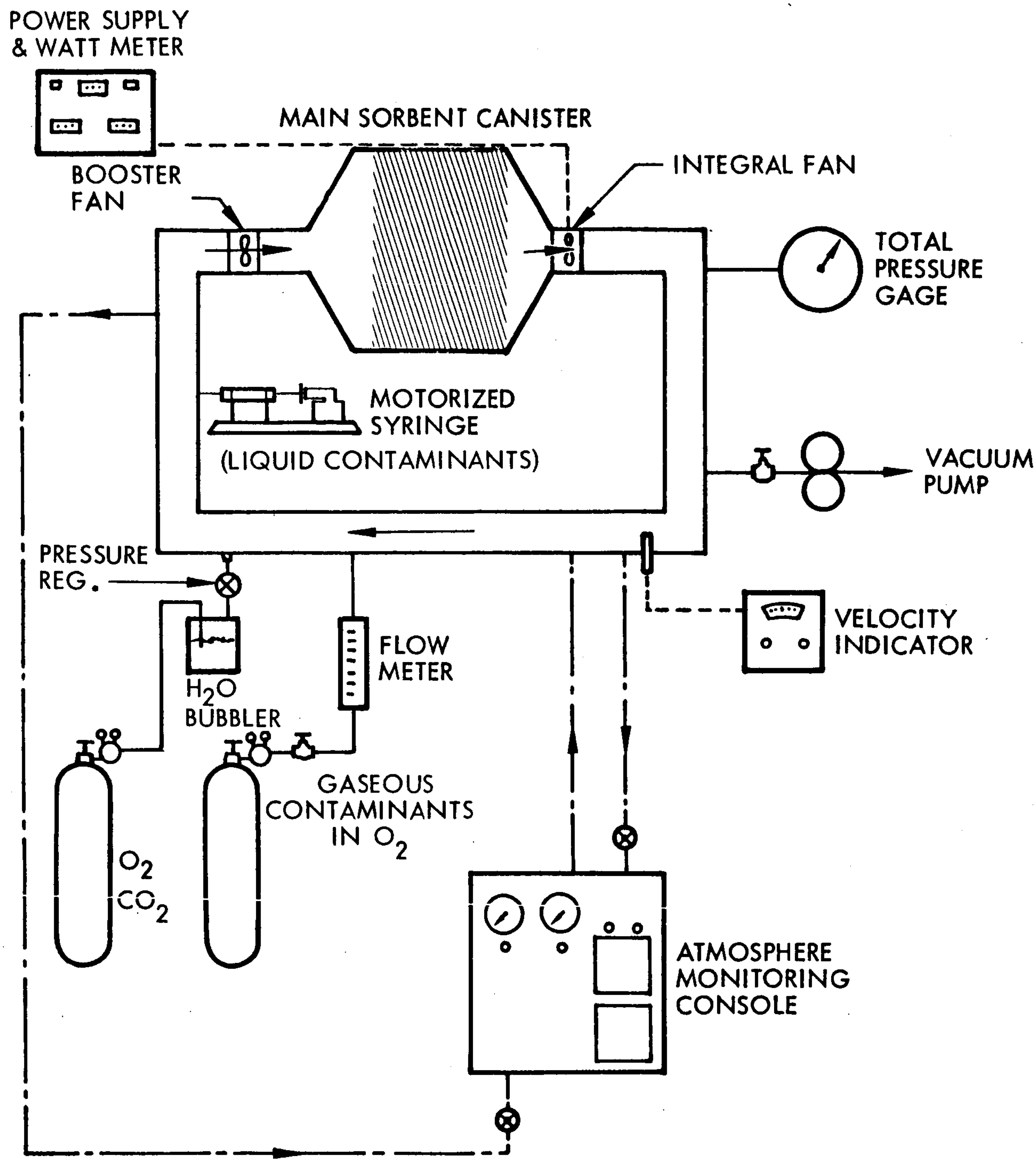

Figure 3-1 Apparatus For Main Sorbent Test

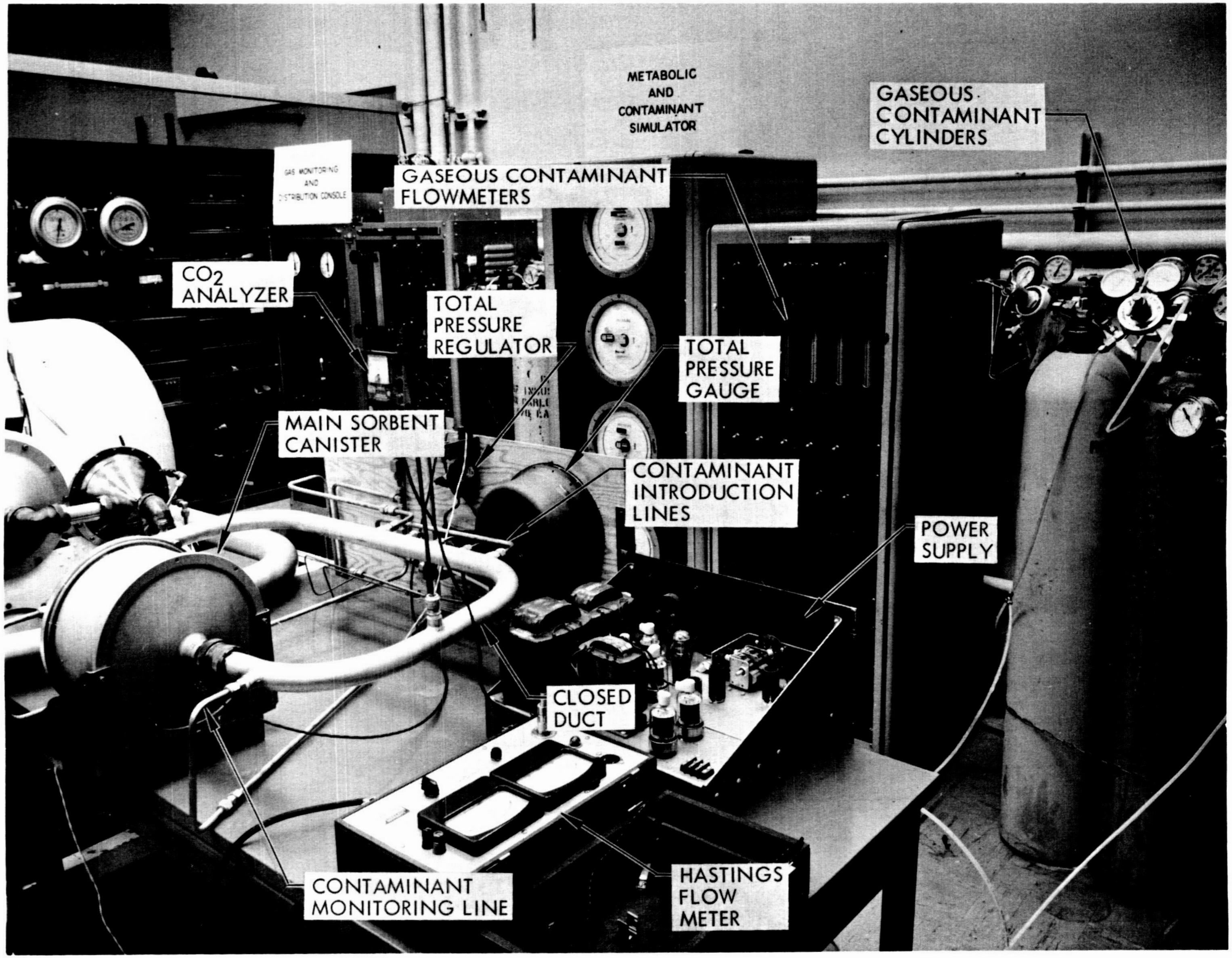

Figure 3-2 Setup For Main Sorbent Bench Test

3.3 PROCEDURE

The main sorbent subsystem tests were performed in the following manner.

3.3.1 Controller Checkout

The main panel power and sorbent fan were energized. Operation of the fan was observed.

3.3.2 Power and Pressure Drop Determination

With the fan energized, and the system at 5 psia, fan power consumption was measured. With the fan energized, and the system at 14.7 psia, pressure drop and flowrate were measured.

3.3.3 Contaminant Removal Capability

With the system operating at design conditions, the contaminants listed in Table 3-1 were introduced at the specified rates. The system was allowed to operate for 24 hours. Inlet and outlet concentrations were measured throughout the test. The sampling point was switched from inlet to outlet every three hours.

After the above measurements were made, sufficient quantities of n-Butane and Freon-12 were injected into the gas stream to cause an "upset".* This was done with the contaminants listed in Table 3-1 still being introduced at a total of 6.989 grams/day. Concentration of the two upset contaminants were monitored at the canister outlet for four hours.

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*Rapid increase to five times "Spacecabin Maximum Allowable Concentration," SMAC.

3.4 RESULTS

3.4.1 Controller Checkout

When the panel power was energized, all of the appropriate lights were illuminated. When the sorbent fan was energized, the sorbent fan operated.

3.4.2 Power and Pressure Drop Determination

The pressure drop of the main sorbent bed was measured as a function of flowrate at 14.7 psia, and the results were corrected to the 5 psia pure oxygen condition. Data for the 5 psia condition are presented in Figure 3-3. The fan pressure rise was calculated from 14.7 psia test data, and measured by the supplier (Globe Industries) for the 5 psia pure oxygen condition. These data are shown in Figure 3-3.

The fan power was measured at 5 psia, and 10 cfm, and determined to be 7.5 watts.

3.4.3 Contaminant Removal Capability

During the test, data were taken on oxygen, carbon dioxide and contaminant concentrations to establish the operating characteristics of the system. These data are presented in Table 3-1 and Figures 3-4 through 3-8.

The oxygen concentration shown in Figure 3-4 equilibrated at 85 percent. Carbon dioxide concentration (Figure 3-5) varied between 7.4 mm Hg and 5.7 mm Hg throughout the test. Data on contaminant concentrations shown in Table 3-1 indicate that none of the contaminants exceeded SMAC. In cases where no contaminants were detected, the concentrations were assumed to be below the instrument sensitivity.

Table 3-1 presents (1) the contaminants introduced, (2) their SMAC values, (3) the estimated instrument sensitivity, (4) the anticipated contaminant introduction rate, (5) the actual contaminant introduction rate, and (6) the contaminant concentration measured after 24 hours of testing.

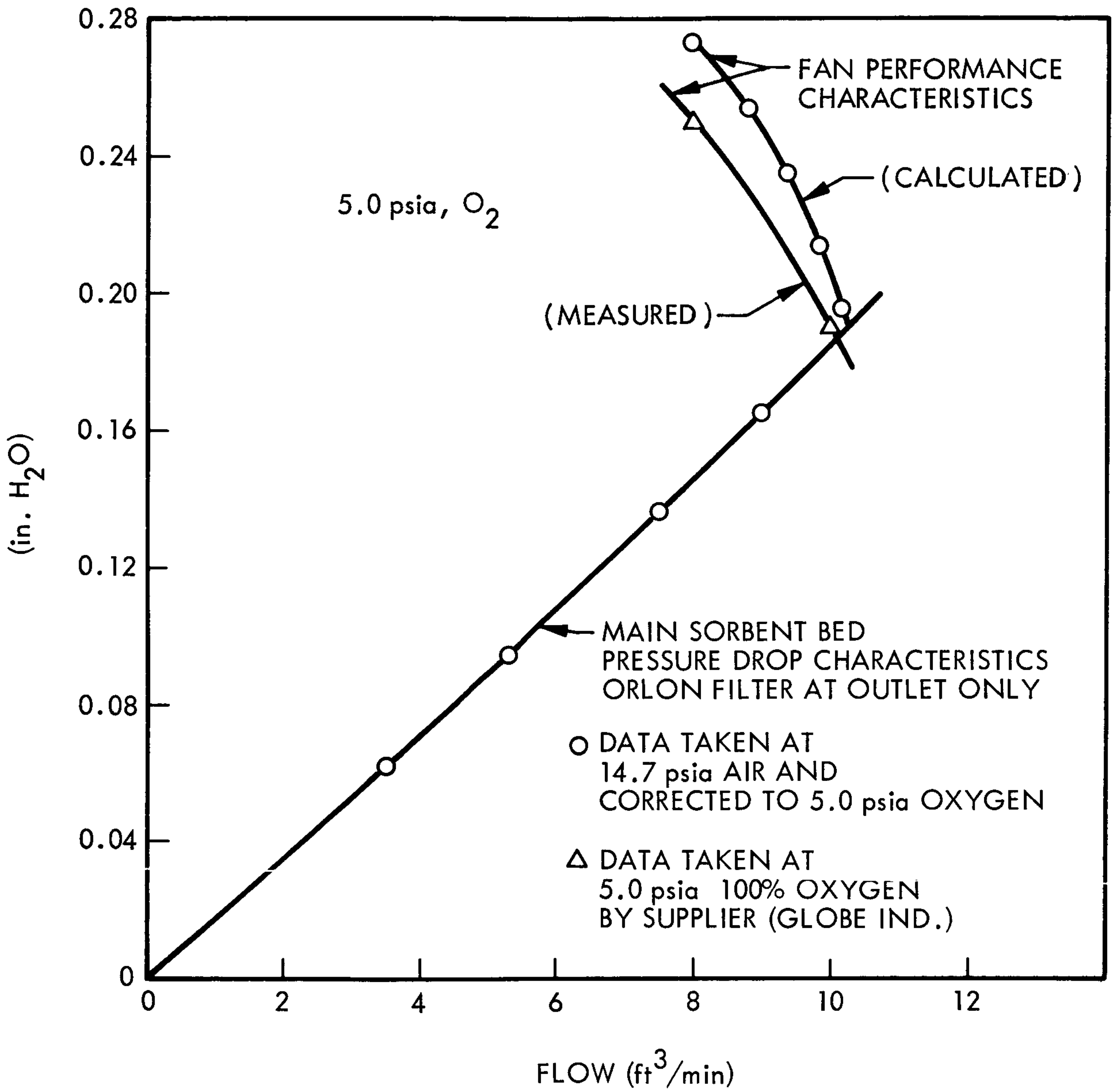

Figure 3-3 Main Sorbent Pressure Drop and Fan Performance

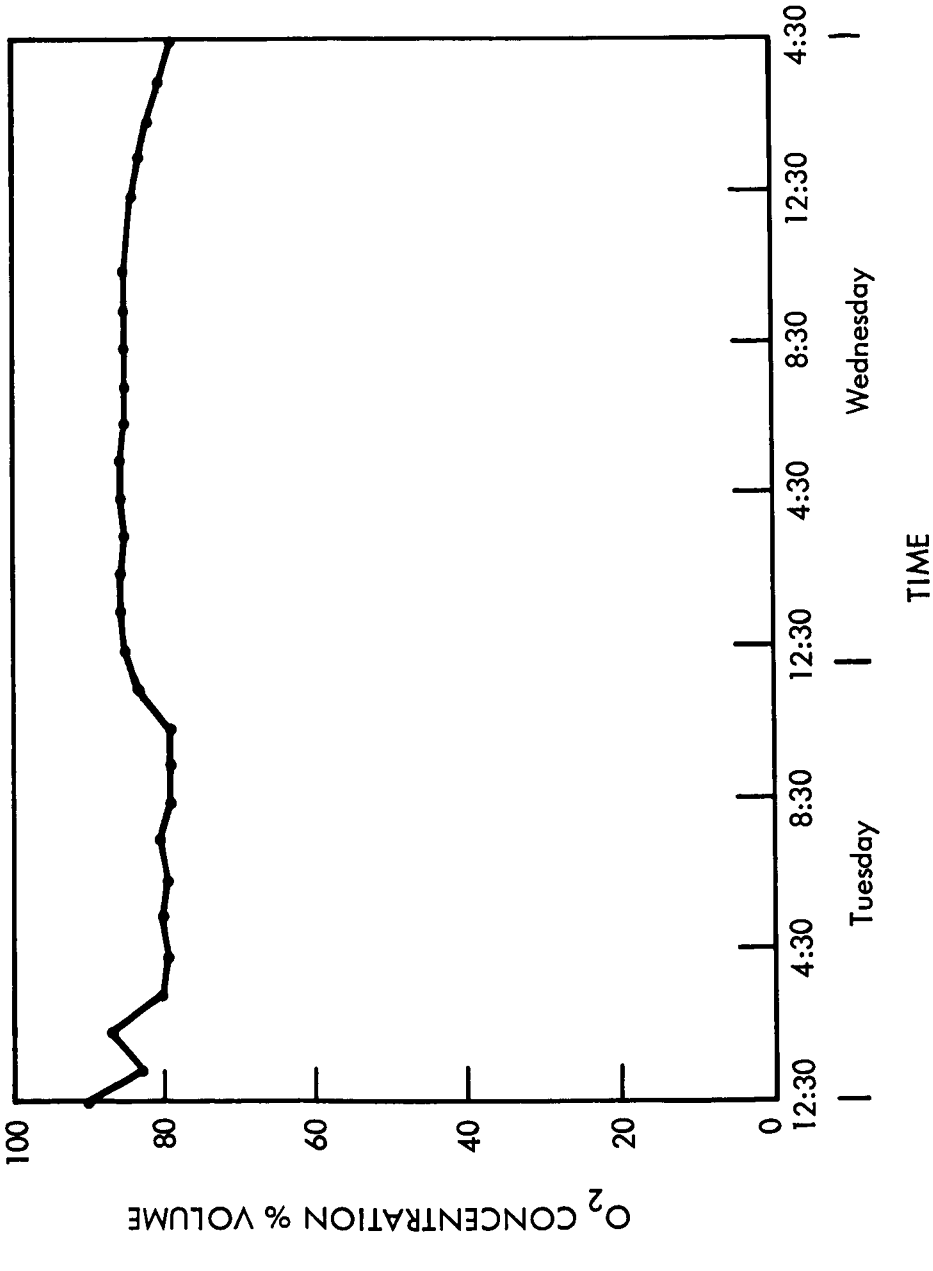

Figure 3-4 Test Results, Main Sorbent Bed, June 8 and 9, 1965, 5 psia

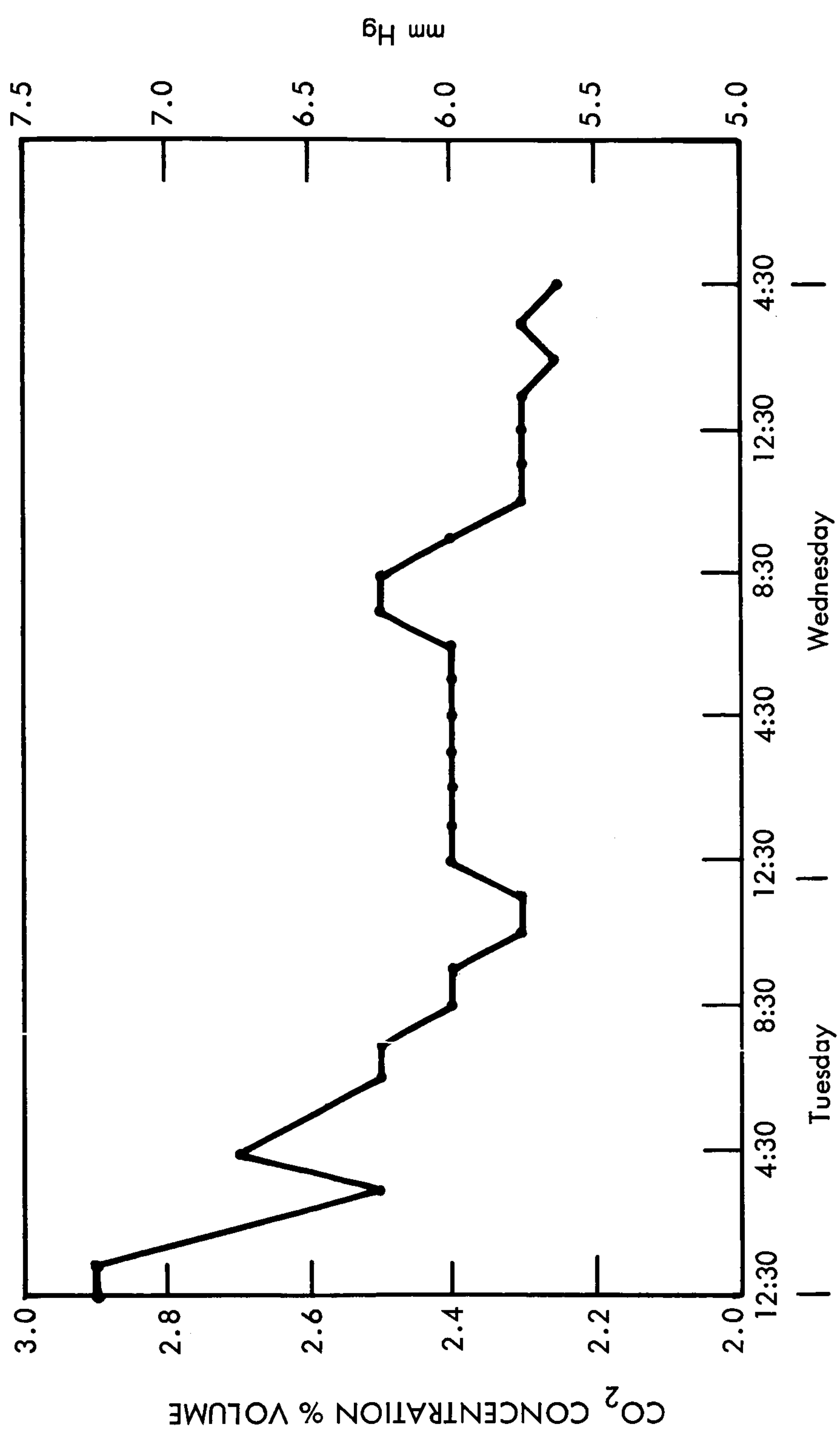

Figure 3-5 Test Results, Main Sorbent Bed, June 8 and 9, 1965, 5 psia

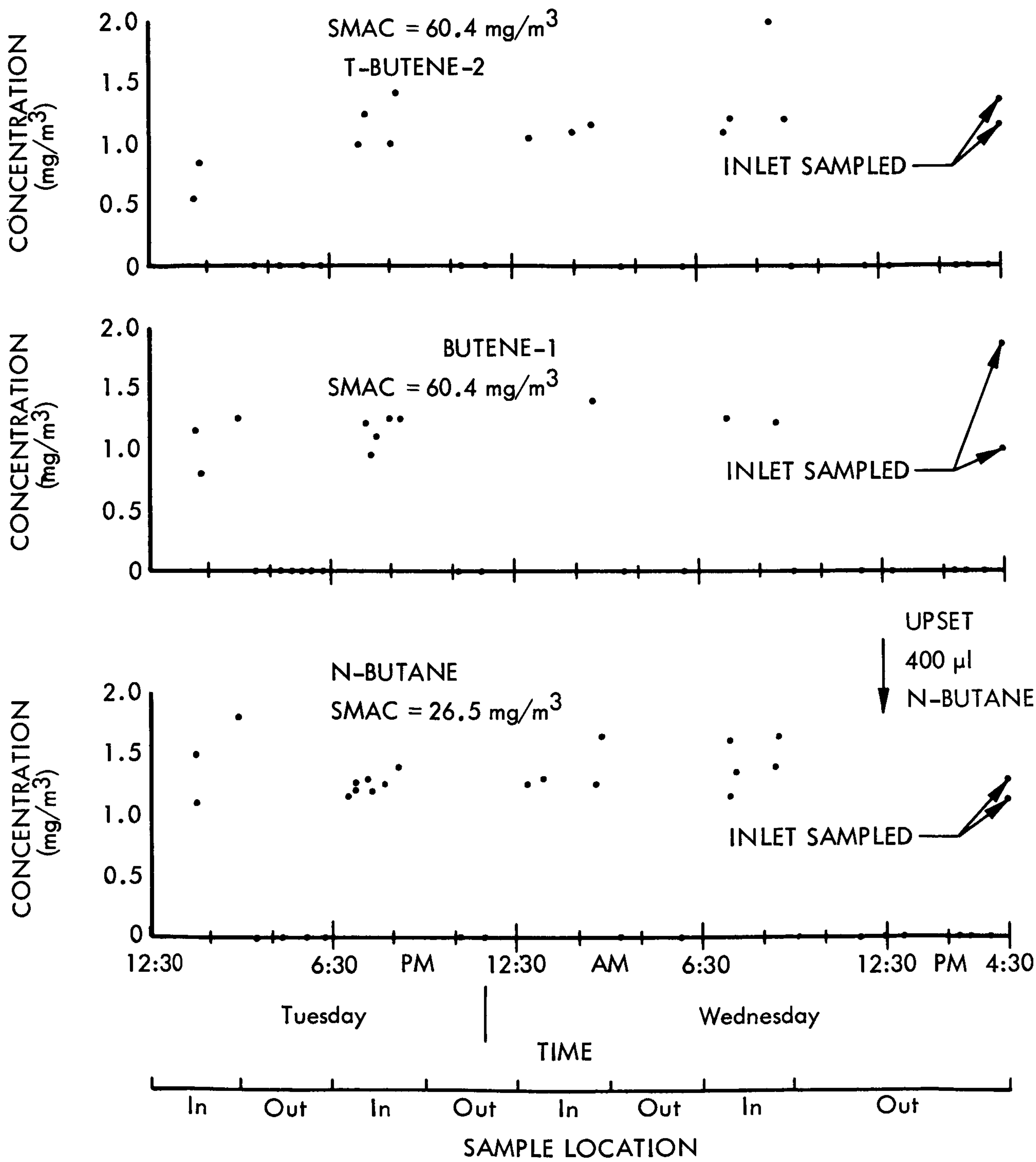

Figure 3-6 Test Results, Main Sorbent Bed, June 8 and 9, 1965, 5 psia

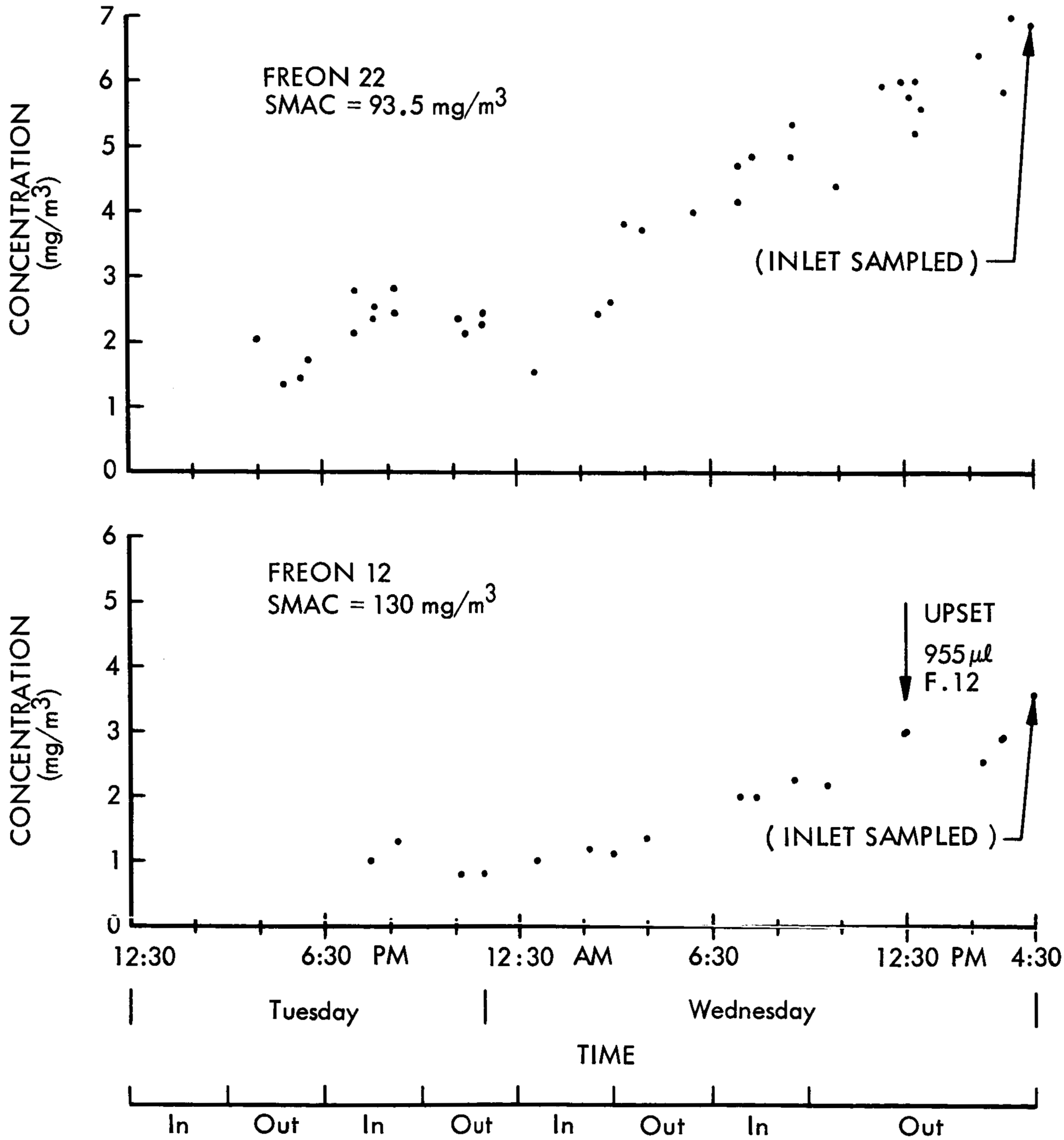

Figure 3-7 Test Results Main Sorbent Bed, June 8 and 9, 1965, 5 psia

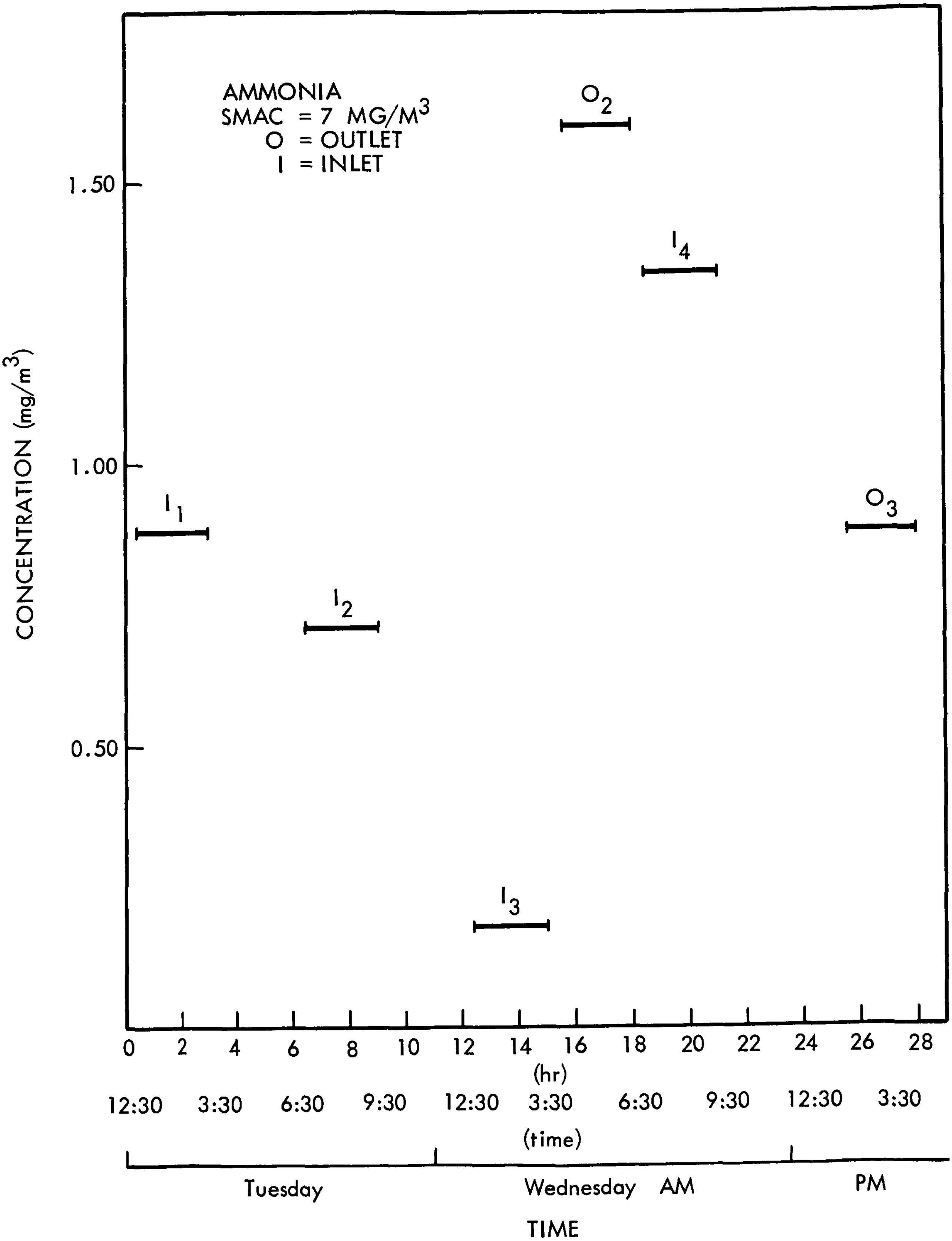

Figure 3-8 Test Results, Main Sorbent Bed, June 8 and 9, 1965, 5 psia, Ammonia

3.5 DISCUSSION

3.5.1 Pressure Drop

The data taken on the main sorbent bed pressure drop characteristics and fan pressure rise indicate that a flow rate of 10.2 cfm occurs at 5 psia, with a pressure drop of 0.19 inches of water.

During the test an additional fan was used to overcome the pressure drop of the test duct. The flow was measured during this portion of the test and maintained at 10 cfm by controlling the speed of the additional fan.

3.5.2 Oxygen and CO_2 Concentration

The oxygen and carbon dioxide concentrations within the system are dependent upon (1) the initial concentration, (2) the inflow leakage to the system, (3) the outflow taken for colorimetric analysis, and (4) the oxygen-carbon dioxide makeup flow rates.

The reason for the oxygen concentration equilibrating at 85%, and for the decay in CO_2 concentration, can be explained by examining the mass balance shown in Figure 3-9. The gas outflow for colorimetric analysis (50 scc/min) and the N_2 concentration (10.5%) are known from test measurements. At equilibrium, the nitrogen leakage inflow must equal the nitrogen withdrawal, which is 5.3 scc/min. Thus the total inflow leakage of air is 6.6 scc/min. Since the oxygen-contaminants mixture is introduced at 20 scc/min, the total pressure regulator admitted 23.4 scc/min (50-26.6) or approximately 1/2 the total amount of gas entering the system. The decay in CO_2 concentration occurred because the 3% CO_2 mixture was admitted through the total pressure regulator only. A similar variation should have occurred in water vapor content, although it was not measured.

The initial concentration of oxygen, CO_2 and water vapor was reached by purging the system with the gas admitted through the total pressure regulator and bubbler.

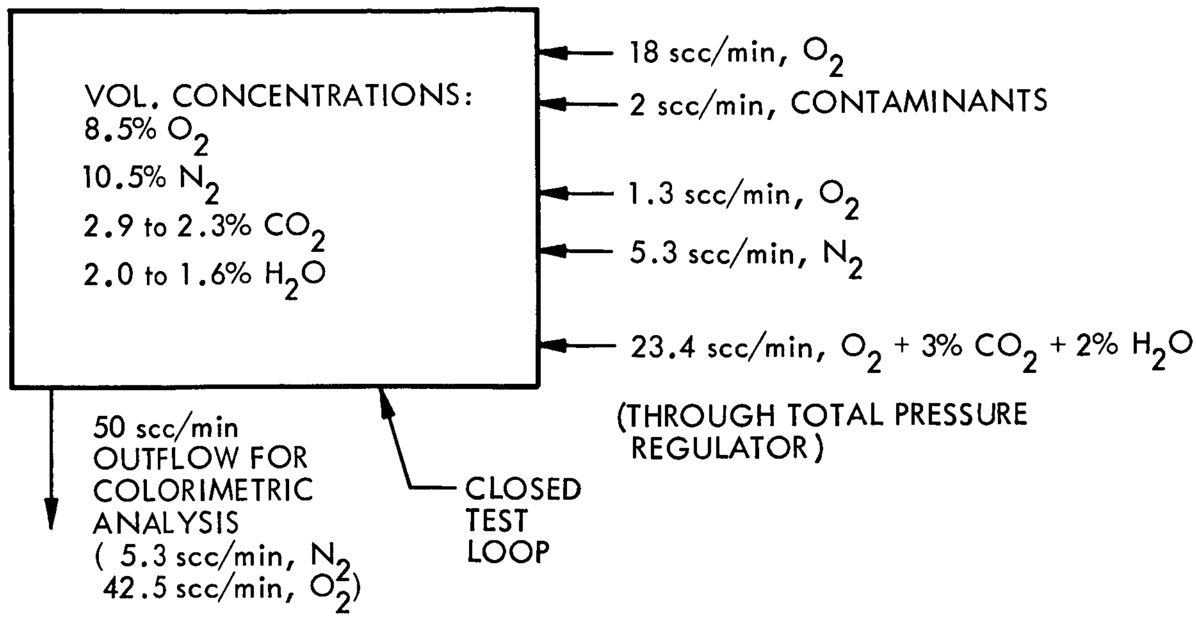

Figure 3-9 Mass Balance for Main Sorbent Test

3.5.3 Contaminant Removal Capability-Normal Conditions

For contaminants with a clear difference between inlet and outlet concentration, which have reached steady state conditions, the removal efficiency η_r can be determined from the following relation.

$$\eta_r = \frac{C_i - C_o}{C_i}$$

where

C_i = inlet concentration

C_o = outlet concentration

This is the case for butene-2, butene-1 and n-Butane.

For contaminants which have not reached steady state conditions, the removal efficiency can be determined from the relation

$$C_\theta = \frac{\dot{m}_{intro}}{F\eta_r}\left(1 - e^{\frac{-F\eta_r\theta}{V}}\right)$$

where

C_θ = concentration at time θ
$\dot{m}_{intro}$ = contaminant introduction rate
F = sorbent bed flow rate
V = total system volume
θ = time

This relationship can be used for Freon 22 and Freon 12.

Applying the above equations, the removal efficiencies shown in Table 3-2 are obtained.

Table 3-2

CONTAMINANT REMOVAL EFFICIENCIES

Contaminant	η_r
n-Butane	1.0
1-Butene	1.0
trans-Butene-2	1.0
Freon 12	0.38
Freon 22	0.08

The data for NH_3 (Figure 3-8) was scattered, with inlet and outlet concentration varying considerably, making it impossible to determine η_r. The only explanation for this appears to be error in the wet chemical colorimetric analysis. Data scatter would be substantially reduced if the third inlet and second outlet data points were reversed. The highest concentration measured, however, (1.6 mg/m^3) is below the prediction of 3.5 mg/m^3.

3.5.4 Contaminant Removal Capability – Upset Conditions

After 24 hours, 955 μl of Freon 12 and 400 μl of n-Butane were injected to simulate an upset condition. This was a sufficient quantity to raise the concentration to five times SMAC. Freon 12 was used in lieu of Freon 11 due to problems encountered in the introduction of Freon 11. No change in contaminant concentration for either n-Butane or Freon 12 was observed following the upset. Since the data sampling rate is once every 15 minutes it can be assumed that the rise and decay in contaminant concentration occurred during that time, indicating rapid clearing of the upset condition.

3.5.5 Comparison With Predicted Performance

Removal Capability-Normal Conditions. A comparison of the predicted contaminant removal rates and the removal rates demonstrated in this test is shown in Table 3-3.

Table 3-3

COMPARISON OF REQUIRED AND DEMONSTRATED CONTAMINANT REMOVAL CAPABILITIES MAIN SORBENT BED

Contaminant	$\dot{M}$ removal demonstrated* (gm/day)	$\dot{M}$ removal required (gm/day)
n-Butane	8.0	3.58
1-Butene	18.2	2.60
trans-Butene-2	18.2	3.36
Freon 12	17.4	1.70
Freon 22	3.2	0.16

*Based on η_r from test and an inlet concentration equal to SMAC

For this comparison the demonstrated test performance was determined by calculating the removal rate that would have occurred with the removal efficiency determined during the test and an inlet concentration equal to SMAC. The required removal efficiency was taken from Ref. 1, p. 7-24, for the main sorbent bed. The demonstrated removal capability equals or exceeds the required removal capability for the contaminants on which sufficient data were available to make such a comparison.

Removal Capability-Upset Conditions. Due to the small volume of the test system ($0.01\ m^3$) the clearing of the upset condition occurred quite rapidly. Predictions indicated that the concentration should return 95%* from the upset value in less than 1 minute. Since the maximum data sampling rate is about once every 15 minutes, it was not possible to observe the rise and decay in contaminant concentration during the upset condition.

*A "95% return" means that the concentration is reduced by 95% of the difference between the upset concentration and the concentration prior to upset.

Section 4
CATALYTIC OXIDIZER–POST SORBENT TESTS

The catalytic oxidizer-post sorbent tests were conducted from July 13 through July 22, 1965. The following section presents the objectives of the test, apparatus and procedures used, and the results obtained.

4.1 OBJECTIVES

The objectives of the test were:

- Checkout operation of the catalytic oxidizer controller including automatic and manual operation
- Determine the overall pressure drop of the catalytic oxidizer and post sorbent canister, at design conditions*
- Determine the power required by the catalytic oxidizer at design conditions
- Determine oxidation efficiency, with the contaminants listed in Table 4-1 under both normal and upset conditions
- Determine the presence of products of combustion and the ability of the post sorbent to remove these compounds

4.2 APPARATUS

The apparatus used in the catalytic oxidizer-post sorbent subsystem tests is shown in Figures 4-1 and 4-2. The test equipment included the following major elements:

- Cylinders for contaminant, oxygen and carbon dioxide supply
- Flow meters to monitor contaminant and total flow rates
- Total pressure gages to measure system pressure
- A total pressure regulator to control system pressure

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*5 psia, t inlet = 130°F, γ = 0.03 $\#H_2O/\#O_2$, $t_{catalyst}$ 785°F, flow = 1 cfm

Table 4-1

CONTAMINANTS INTRODUCED DURING CATALYTIC BURNER-POST SORBENT SUBSYSTEM TESTS

Oxidation Efficiency Test

Contaminants Introduced	Contaminants Monitored
Carbon Monoxide	All contaminants introduced
Acetylene	
Formaldehyde	
Methyl Mercaptan	
Propylene	
Methane	
Hydrogen	

Products of Oxidation Test

Contaminants Introduced	Contaminants Monitored
Freon-114	All contaminants introduced and:
Hydrogen Sulfide	Hydrogen Fluoride*
Dimethyl Hydrazine	Phosgene*
	Hydrochloric Acid*
	Sulfur Dioxide*
	Nitrogen Dioxide*

*Monitored at catalytic oxidizer and post sorbent exit only

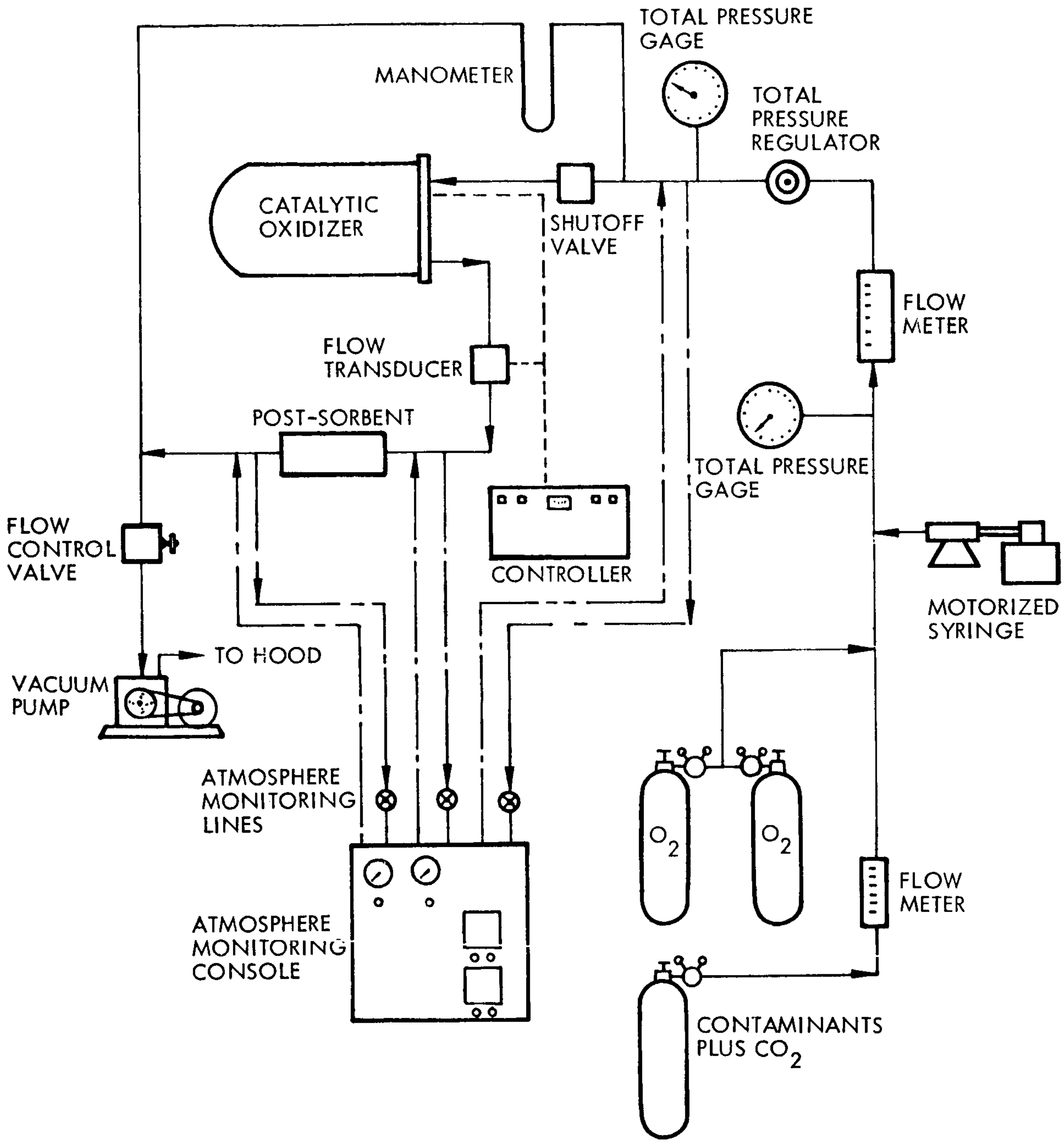

Figure 4-1 Test Apparatus For Catalytic-Oxidizer/Post-Sorbent Subsystem

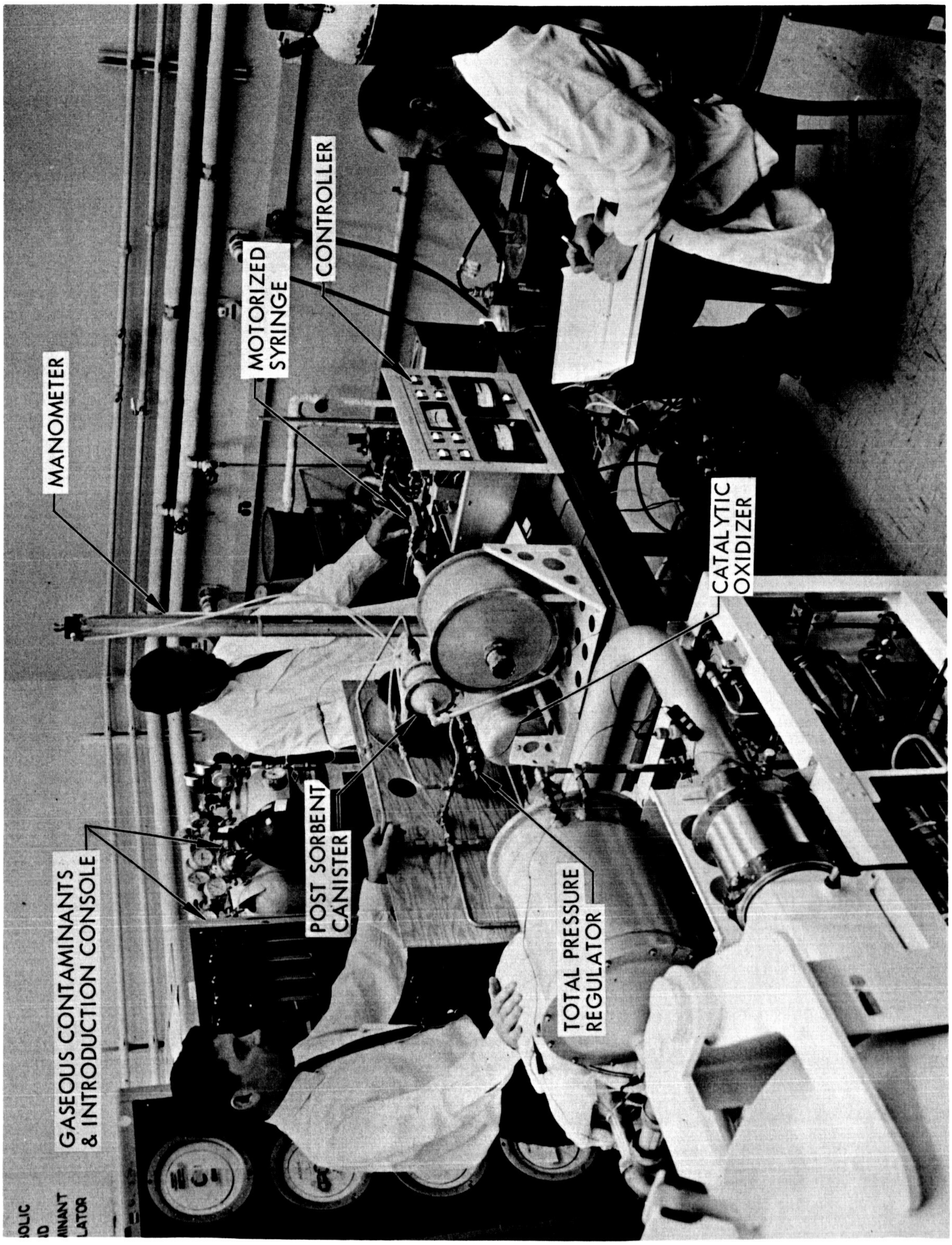

Figure 4-2 Setup For Catalytic Oxidizer – Post Sorbent Test

- A manometer to determine pressure drop across the catalytic oxidizer-post sorbent unit
- A vacuum pump and flow control valve to control system flow rate (part of atmosphere monitoring console)
- An atmosphere monitoring facility to perform gas analysis
- Voltmeter and ammeter to determine power consumption

4.3 PROCEDURE

The catalytic oxidizer-post sorbent subsystem tests were performed in the following manner.

4.3.1 Controller Checkout

The controller was energized, and operation of the automatic heater control and shutoff valve control were verified. Manual heater operation and manual shutoff valve operation were then verified. Oxygen flow was established at design conditions and the flow meter reading on the controller was verified.

4.3.2 Power and Pressure Drop Determination

With the controller energized in the automatic mode and the system at design conditions, the system pressure drop and power consumption were measured.

4.3.3 Oxidation Efficiency Test

With the system operating at design conditions, the contaminants listed in Table 4-1 (Oxidation Efficiency Test), were introduced at the system inlet. This was done for normal and upset conditions. For each of these conditions the system was allowed to stabilize for several hours before inlet and outlet contaminant concentrations were measured.

4.3.4 Products of Oxidation Test

With the system operating at design conditions, the contaminants listed in Table 4-1 (Products of Oxidation Test) were introduced at the system inlet. This was done for normal conditions only. The system was allowed to stabilize for several hours and then contaminant concentrations were measured at the catalytic oxidizer inlet, catalytic oxidizer outlet, and post sorbent outlet.

4.4 RESULTS

4.4.1 Controller Checkout

The controller checkout was performed as outlined in the procedure and all operations were successful.

4.4.2 Power and Pressure Drop Determination

During the test, the pressure drop characteristic of the catalytic oxidizer-post sorbent subsystem was measured and is shown in Figure 4-3. This data includes the pressure drop of the shutoff valve, the catalytic oxidizer, the flow transducer, the post-sorbent bed and the interconnecting plumbing. The unit was operating at 5.0 psia with oxygen. The catalyst bed temperature was 785°F.

The power consumption for the catalytic oxidizer was measured and determined to be 90 watts average. Peak power is 160 watts with a duty cycle of 55 percent on. At design conditions the duty cycle is typically 7.0 minutes on, and 5.7 minutes off.

4.4.3 Oxidation Efficiency Test

The results of the oxidation efficiency test are shown in Table 4-2. This table lists the contaminants introduced, their SMAC values, the desired contaminant inlet concentration, the instrumentation sensitivity, the measured inlet and outlet concentrations for normal and upset conditions, and the removal efficiencies for normal and upset

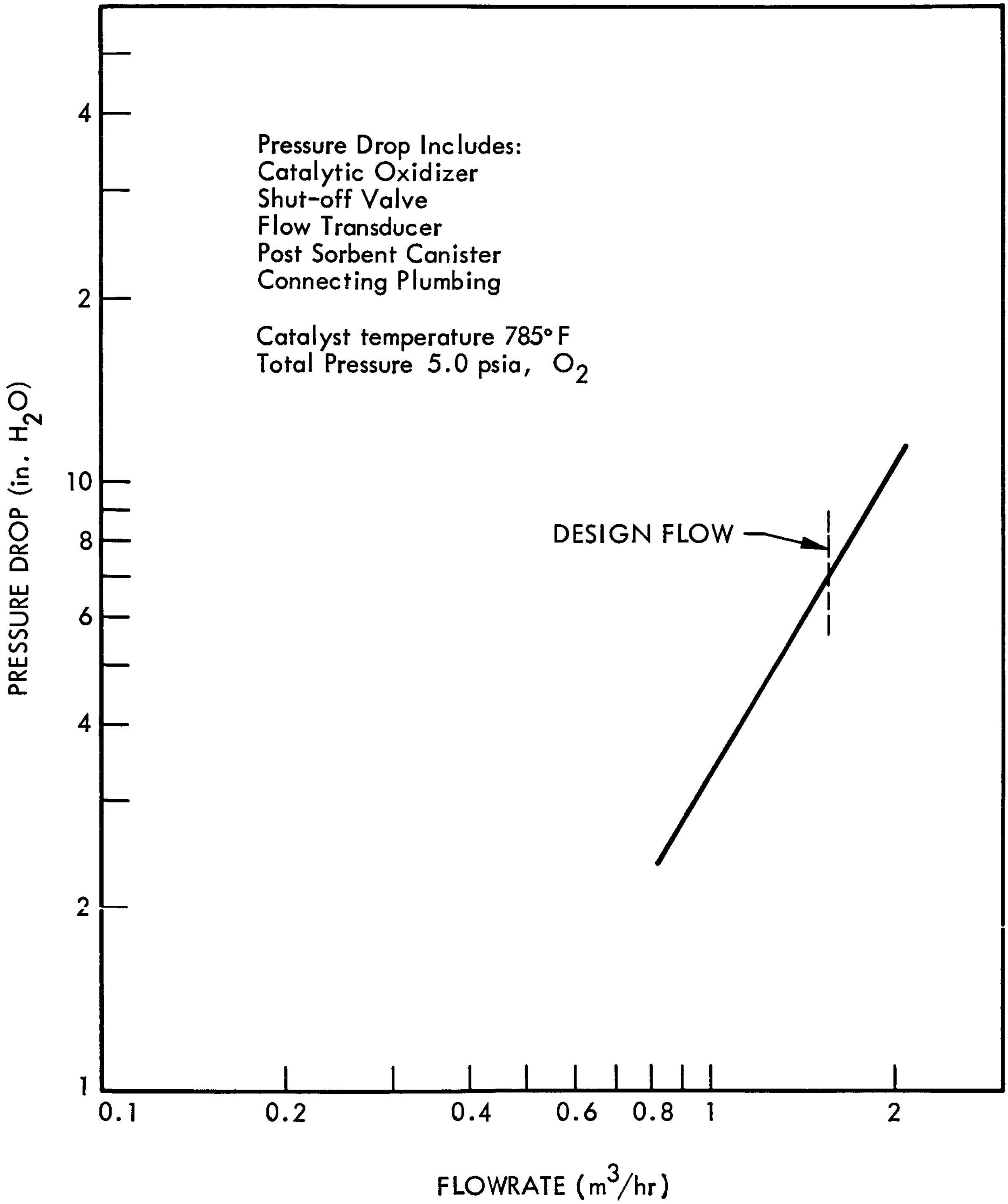

Figure 4-3 Pressure Drop Versus Flow Catalytic Oxidizer—Post Sorbent

Table 4-2

OXIDATION EFFICIENCY TEST RESULTS

Contaminant	SMAC (mg/m^3)	Desired Inlet Concentration (mg/m^3)	Instrument Sensitivity (mg/m^3)	Inlet Concentration (mg/m^3)		Outlet Concentration (mg/m^3)		Removal Efficiency (%)	
				Normal	Upset	Normal	Upset	Normal	Upset
Carbon Monoxide	22	69	6.9	59.5	162.0	12.6	26.6	79	83
Acetylene	28	28	0.9	35.8	107.0	0.96	3.19	97	97
Methyl Mercaptan	8	8	0.5	1.3	4.4	< 0.5	1.20	> 62	74
Propylene	45	45	0.2	20.9	62.3	1.12	4.70	95	93
Methane	1720	860	17.0	670.0	1930.0	125.0	376.0	81	81
Hydrogen	215	107	3.6	167.0	404.0	31.4	31.4	83	92
Formaldehyde	1	5	0.5	1.62/1.12*	2.37/1.45*	2.26/1.82*	1.81/2.64*	**	**

*Formaldehyde data taken colorimetrically with two data points per condition
**Data scatter prevented calculation

conditions. All of these data were taken with the catalyst bed at 785°F, the flow at 1 cfm and the total pressure 5 psia, in an oxygen atmosphere.

4.4.4 Products of Oxidation Test

The results of the products of oxidation study are shown in Table 4-3. This table lists the contaminants introduced and monitored, their SMAC values, the instrument sensitivity, the desired contaminant inlet concentration and the measured inlet and outlet concentrations. The test was performed for normal conditions with the catalyst bed at 785°F, the flow at 1 cfm, the total pressure at 5 psia, in an oxygen atmosphere.

4.5 DISCUSSION

4.5.1 Oxidation Efficiency Test

Inlet Concentration. The desired contaminant inlet concentrations under normal conditions were SMAC, or 10 times the instrumentation sensitivity, whichever was greater, except for the case of methane and hydrogen. For methane and hydrogen, the desired inlet concentration during normal conditions was to result in a total concentration of these two contaminants of 2.0 mm Hg. For upset conditions, the inlet concentrations were to be five times the values used under normal conditions for all contaminants except hydrogen and methane; for these, upset equals 2.5 times SMAC.

The method for establishing inlet contaminant concentrations was to inject contaminants at known rates in the 1 cfm flow stream. Gaseous contaminants were mixed in cylinders in dilute form and metered into the stream through a flow meter. Liquid contaminants were injected through a motorized syringe.

Differences between the desired and measured inlet concentrations were caused either by errors in the concentrations of contaminants in the mixture or errors in flow measurement. Knowledge of the inlet concentration, rather than precise control of it, was the primary objective.

Table 4-3

PRODUCTS OF OXIDATION TEST RESULTS

Contaminant	SMAC (mg/m^3)	Instrument Sensitivity (mg/m^3)	Desired Inlet Concentration (mg/m^3)	Catalytic Oxidizer Inlet (mg/m^3)	Catalytic Oxidizer Exit (mg/m^3)	Post Sorbent Exit (mg/m^3)
Freon 114	184.0	0.3	83.0	161.0	34.0	34.0
Hydrogen Sulfide	6.0	0.05	6.0	14.0	0.7	0.3
Monomethyl Hydrazine	49.6	10.0	22.5	21.2	0	0
Sulfur Dioxide	2.6	0.15	*	0	0.35	0
Nitrogen Dioxide	0.9	0.02	*	0	0.02	0
Hydrogen Fluoride	0.4	0.04	*	0	0	0
Hydrogen Chloride	1.4	0.20	*	0	0	0
Phosgene	0.08	0.10	*	0	0	0

*Not introduced

Removal Efficiency. The removal efficiencies (η_r) shown in Table 4-2 were determined from the relationship

$$\eta_r = \frac{C_i - C_o}{C_i}$$

where

C_i = contaminant inlet concentration

C_o = contaminant outlet concentration

Little difference occurred in η_r between normal and upset conditions for all contaminants except methyl mercaptan and hydrogen. For methyl mercaptan the calculated removal efficiency for the normal condition could be somewhat high due to instrument sensitivity and the extremely low concentration present at the outlet. Since nothing was detected at the outlet, the calculated η_r was 100 percent. If 0.2 mg/m^3 (well below instrument sensitivity) had been present, η_r would have been 85 percent.

Removal efficiency for formaldehyde was not calculated because the reproducibility of the two colorimetric measurements taken at each condition was poor; the reason for this was not determined.

4.5.2 Products of Oxidation Test

Inlet Concentration. The desired contaminant inlet concentration was based on obtaining a total inlet concentration of 0.02 mm Hg. The total inlet concentration was greater due to an error in setting the flow of the gas mixture containing Freon-114 and hydrogen sulfide. Monomethyl hydrazine was introduced with the motorized syringe, and the measured inlet concentration agreed with the predicted value. Due to the conservative nature of the error, the higher inlet concentrations were accepted.

Formation of Combustion Products. The data shown in Table 4-3 indicate that all three compounds injected (Freon 114, hydrogen sulfide and monomethyl hydrazine) were oxidized. Consequently, the products created should include halogen compounds, sulfur compounds and nitrogen compounds. The only products detected were sulfur dioxide (0.35 mg/m^3) and nitrogen dioxide (0.02 mg/m^3).

In view of the sizeable quantities of Freon 114, hydrogen sulfide and monomethyl hydrazine oxidized, greater quantities of combustion products would be expected at the oxidizer exit. The absence of detectable quantities of HCl and HF is attributed to an acid-base reaction with monomethyl hydrazine.

Removal of Combustion Products. The post sorbent canister removed the sulfur dioxide and nitrogen dioxide; both of these contaminants were absent at the canister exit.

4.5.3 Comparison with Predicted Performance

Pressure Drop. The measured pressure drop at design conditions of the complete catalytic oxidizer post sorbent subsystem, including the flow transducer, was 7.0 inches of water. The pressure drop of the flow transducer, which is test equipment and would not be a part of the flight hardware, is 1.5 inches of water. The available pressure drop for this unit is 7.2 inches of water, based upon installing the unit in the Apollo environmental control system between the lithium hydroxide canister exit and compressor inlet (the location recommended at the conclusion of Phase I).

The pressure drop of the unit, under design conditions (not including the flow transducer) therefore is 1.7 inches less than that available.

Power Consumption. The predicted power consumption for this unit at design conditions was 45 watts. This was based upon an anticipated heat exchanger effectiveness of 90 percent and an overall thermal resistance between the catalyst bed and the outer case of 5.5 Hr °F/BTU. Under these conditions, flow stream heating would account for 7.5 watts, and heat dissipation to ambient would account for 37.5 watts.

The measured power consumption was 90 watts, of which 16.5 watts occurred as flow stream heating (actual heat exchanger effectiveness of 83 percent) and 73.5 watts as heat dissipation to ambient through the insulation and supports.

The increased heat dissipation through the insulation and supports is attributed to discrepancies between the thermal conductivities of the insulating materials used in fabrication and the thermal conductivities assumed in the design analysis. These discrepancies are indicated below.

Material	Design Conductivity (BTU/hr-ft^2-°F/in. at 0.1 mm Hg, 450°F)	Actual Conductivity (BTU/hr-ft^2-°F/in. at 0.1 mm Hg, 450°F)
Taylor Products Molding Material PA-6	3.0	5.5
Owens Corning Heat Felted Fiberglass AA-Fiber	0.04	0.10

To compensate for these increased conductivity values, the conduction path lengths must be increased, and/or cross-sectional areas reduced. This could be achieved by increasing the weight and volume of the unit.

Oxidation Efficiency. The predicted removal efficiency for the contaminants used in this test was 100 percent for all compounds except CH_4 which was 25 percent. These predictions were based upon single-contaminant experiments conducted during Phase I of this program. During these tests, it was determined that the 1.0 percent Pt - 1.0 percent Pd catalyst oxidized CO at 100 percent and CH_4 at 25 percent, per pass, with a catalyst temperature of 785°F and design space velocity. The oxidation efficiencies for the remaining contaminants were predicted to be 100 percent based on the theoretical work done during the contaminant classification study.

As shown in Table 4-2, the measured efficiency during this test for contaminants other than methane was lower than predicted. This decrease in efficiency is attributed to competition between the contaminants present for the active sites on the catalyst. As indicated above, the performance predictions were based upon single-contaminant experiments.

The increase in methane oxidation efficiency is attributed to a difference in bed temperature between this test and earlier tests where η_r was determined to be 25 percent.

A 20-percent increase in flow through the unit will compensate for the removal efficiency decrement experienced.

Section 5
COMPLETE SYSTEM CHAMBER TEST

5.1 OBJECTIVES

The objective of this test was to determine the performance characteristics of the complete trace contaminant removal system (catalytic oxidizer, post sorbent, and main sorbent) in a 200 ft^3 volume, integrated with an Apollo-type environmental control system.

5.2 APPARATUS

The apparatus used is shown in Figures 5-1 through 5-10; it included the following major elements:

- LMSC Two-Gas Regenerative Life Support System to provide thermal control, humidity control and to simulate Apollo contaminant removal via lithium hydroxide, charcoal, and water condensation, Figure 5-1
- Consoles to operate the Two-Gas Regenerative Life Support System and to introduce water-soluble contaminants, Figure 5-2
- Altitude chamber with 200 ft^3 lock to provide Apollo free volume simulation, Figure 5-3
- Contaminant introduction apparatus to introduce gaseous and liquid (water-insoluble) contaminants, Figures 5-4 and 5-5
- Atmosphere Monitoring and Distribution Console to direct chamber gas to analytical instrumentation, Figure 5-6
- Gas chromatographs for atmosphere monitoring, Figures 5-7 and 5-8
- Bubblers used for colormetric gas analysis, Figure 5-9

The general arrangement of this equipment is shown schematically in Figure 5-10.

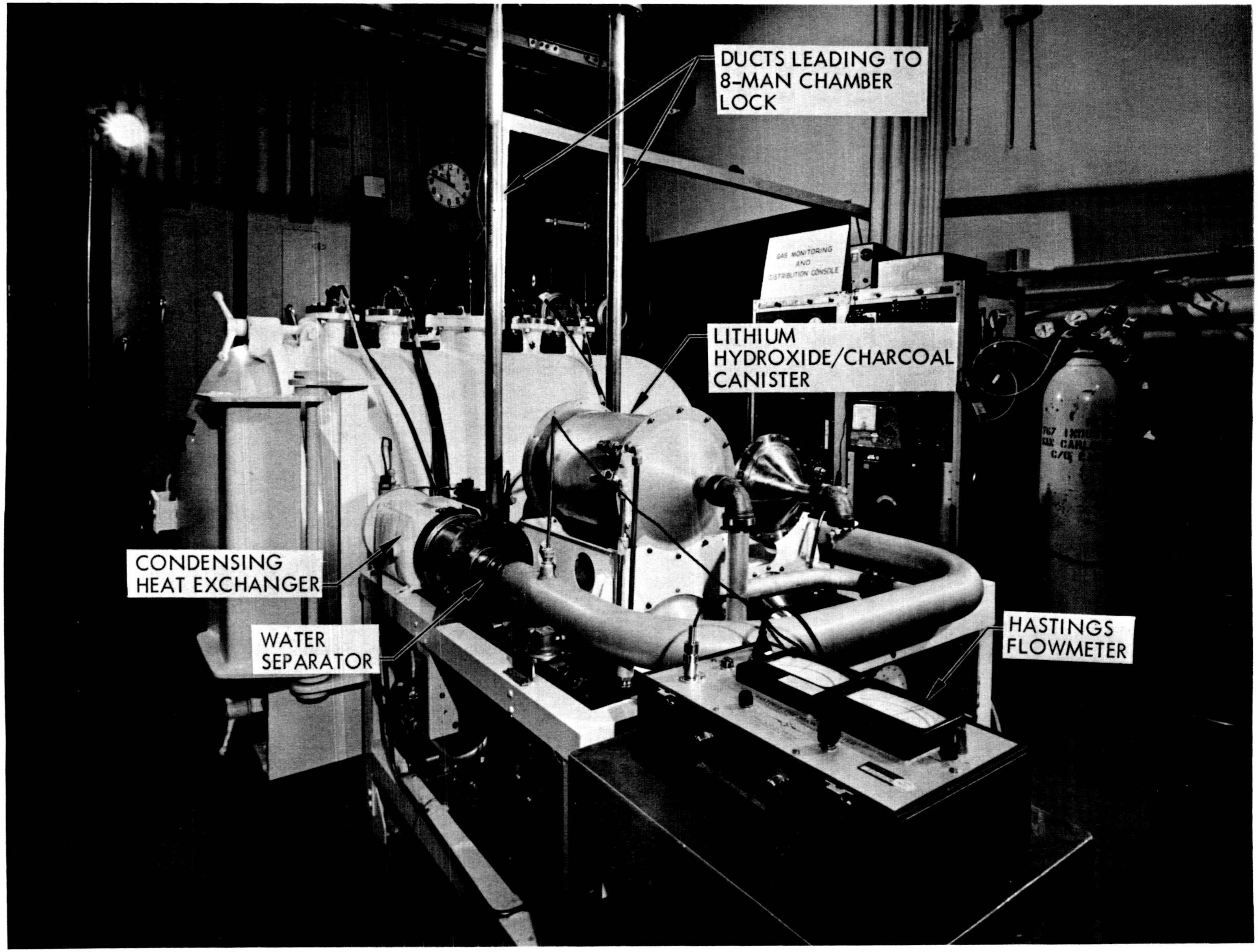

Figure 5-1 Two-Gas Life Support System Modified to Apollo ECS Configuration

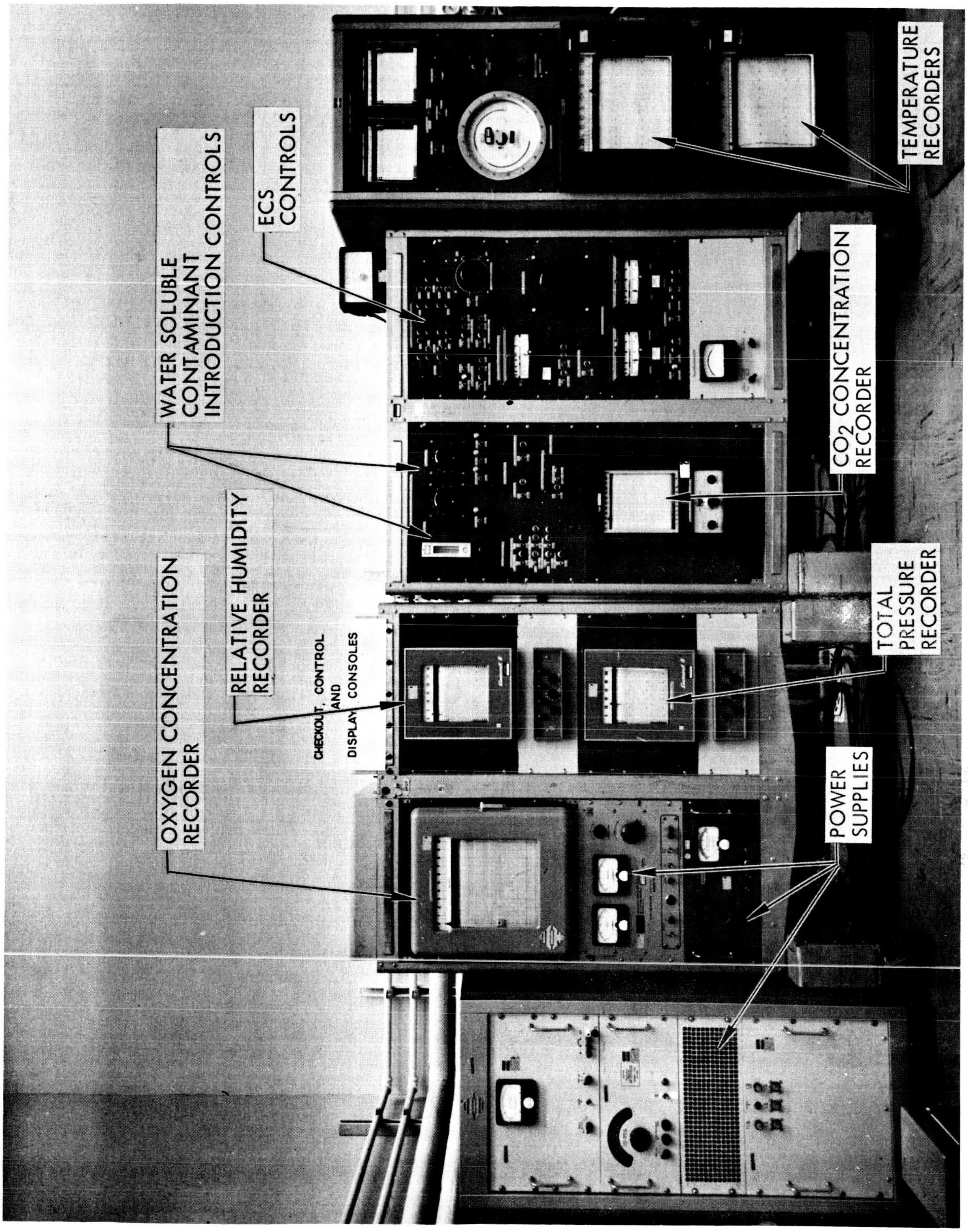

Figure 5-2 Two-Gas Life Support System Operating Consoles

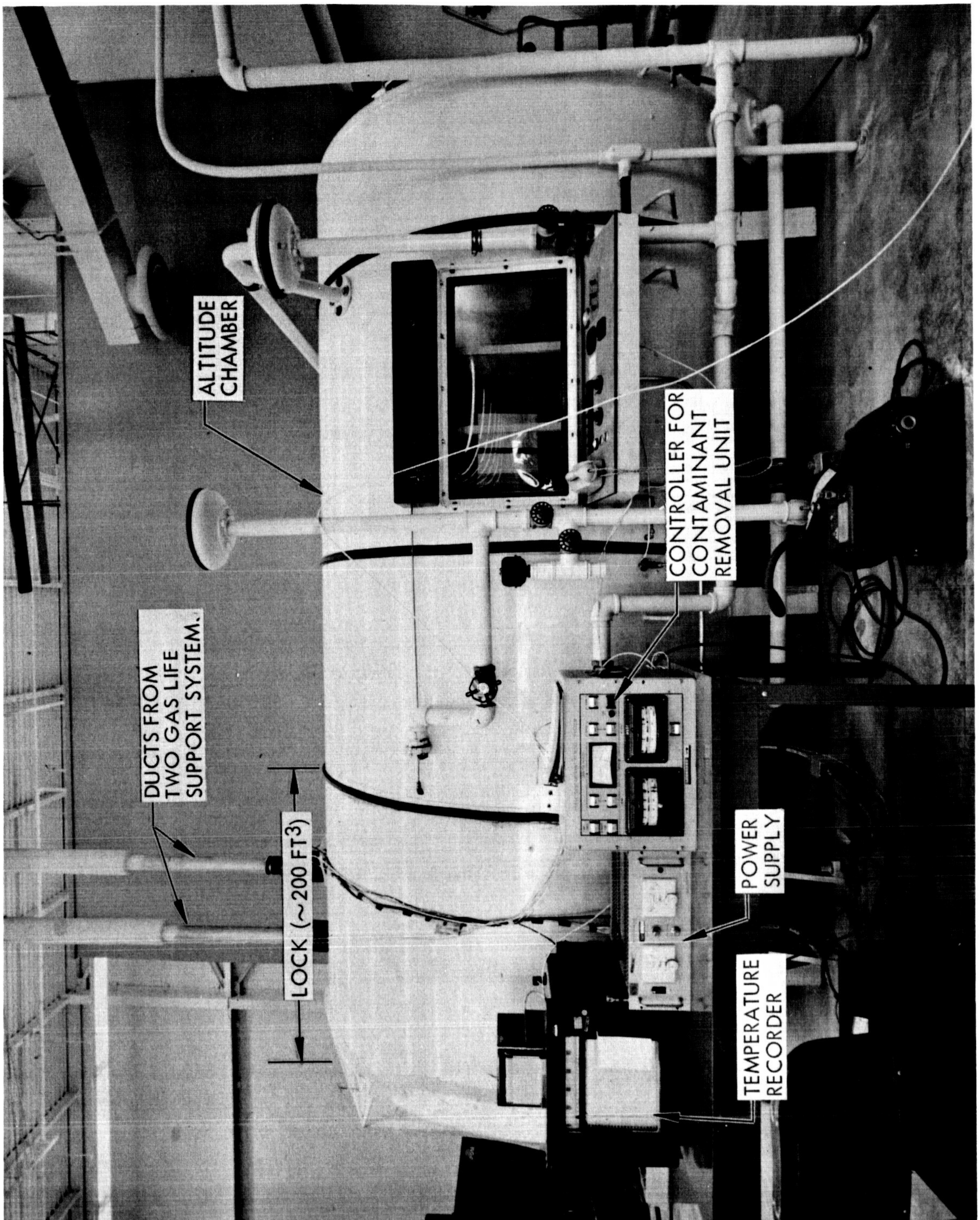

Figure 5-3 Eight Man Chamber With Test Equipment

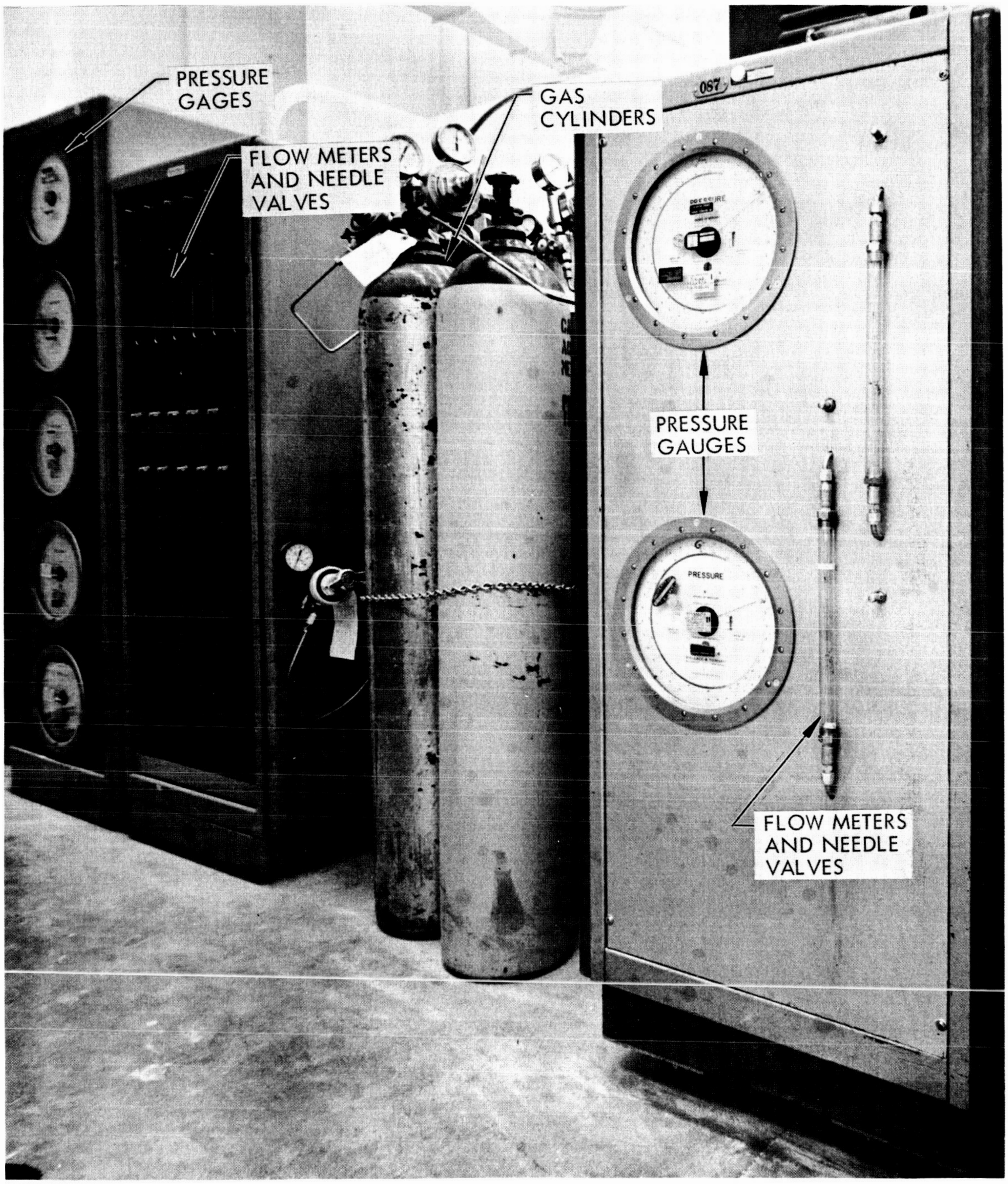

Figure 5-4 Gaseous Contaminant Introduction Console

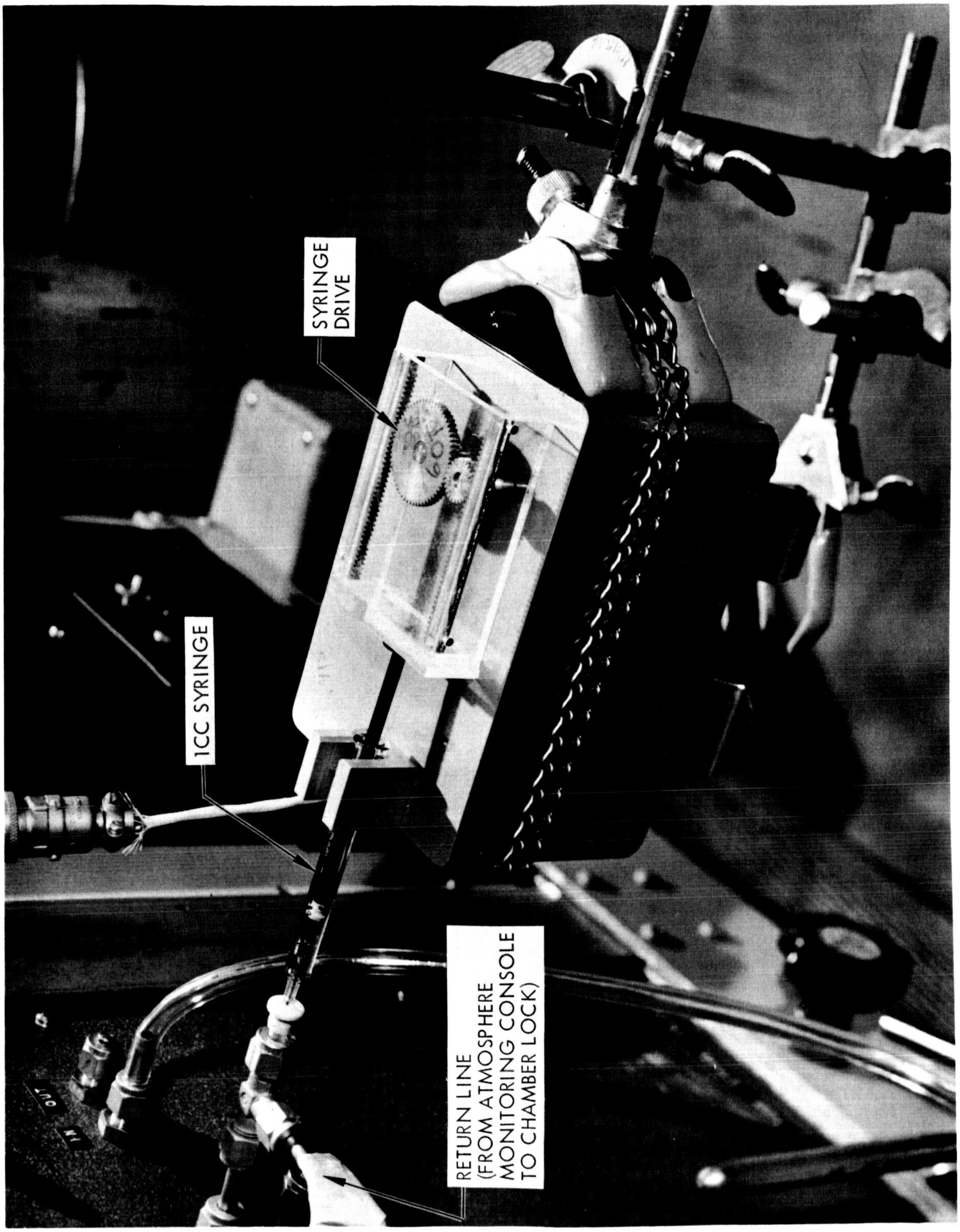

Figure 5-5 Water Insoluble Liquid Contaminant Introduction System

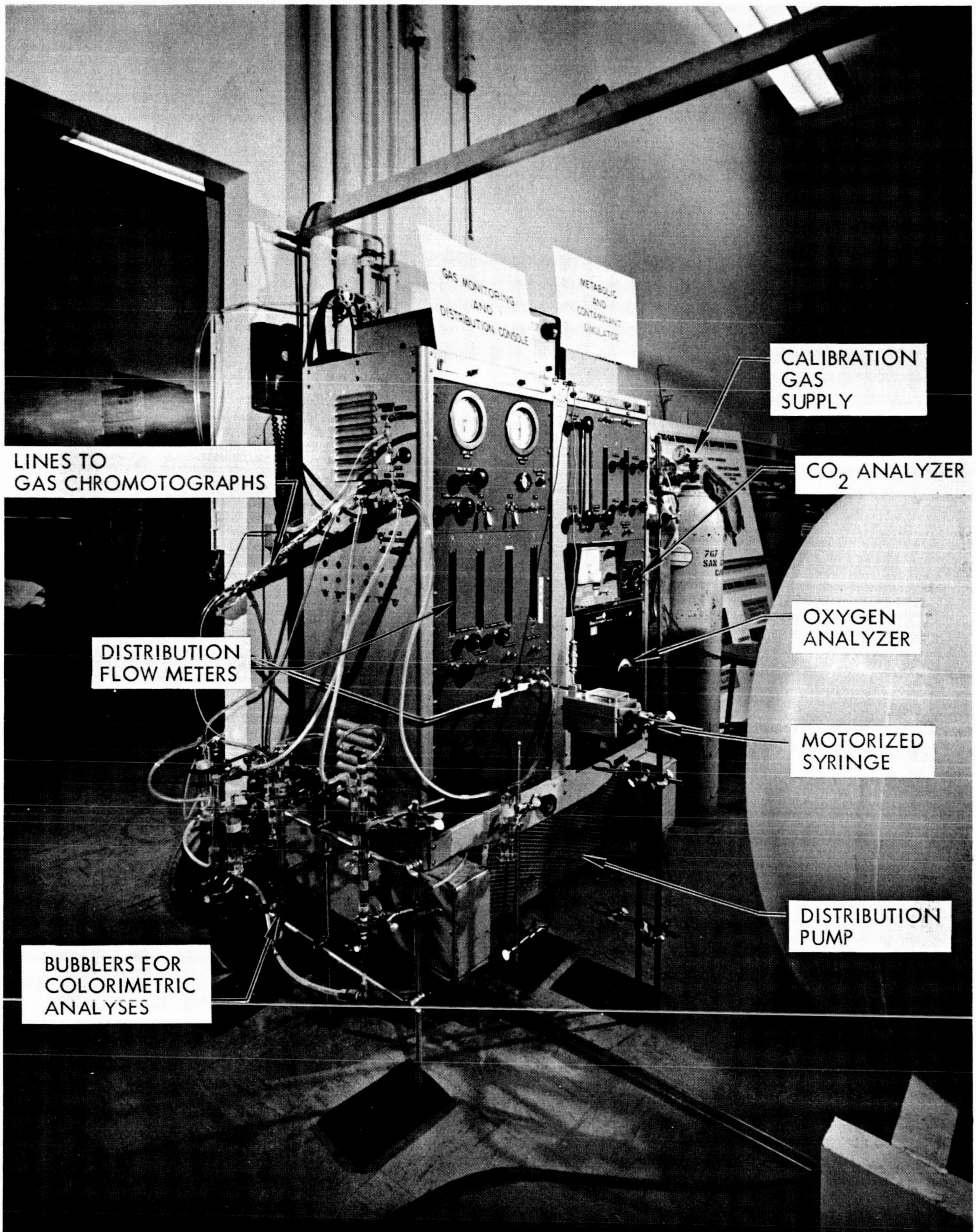

Figure 5-6 Atmosphere Monitoring Distribution Console

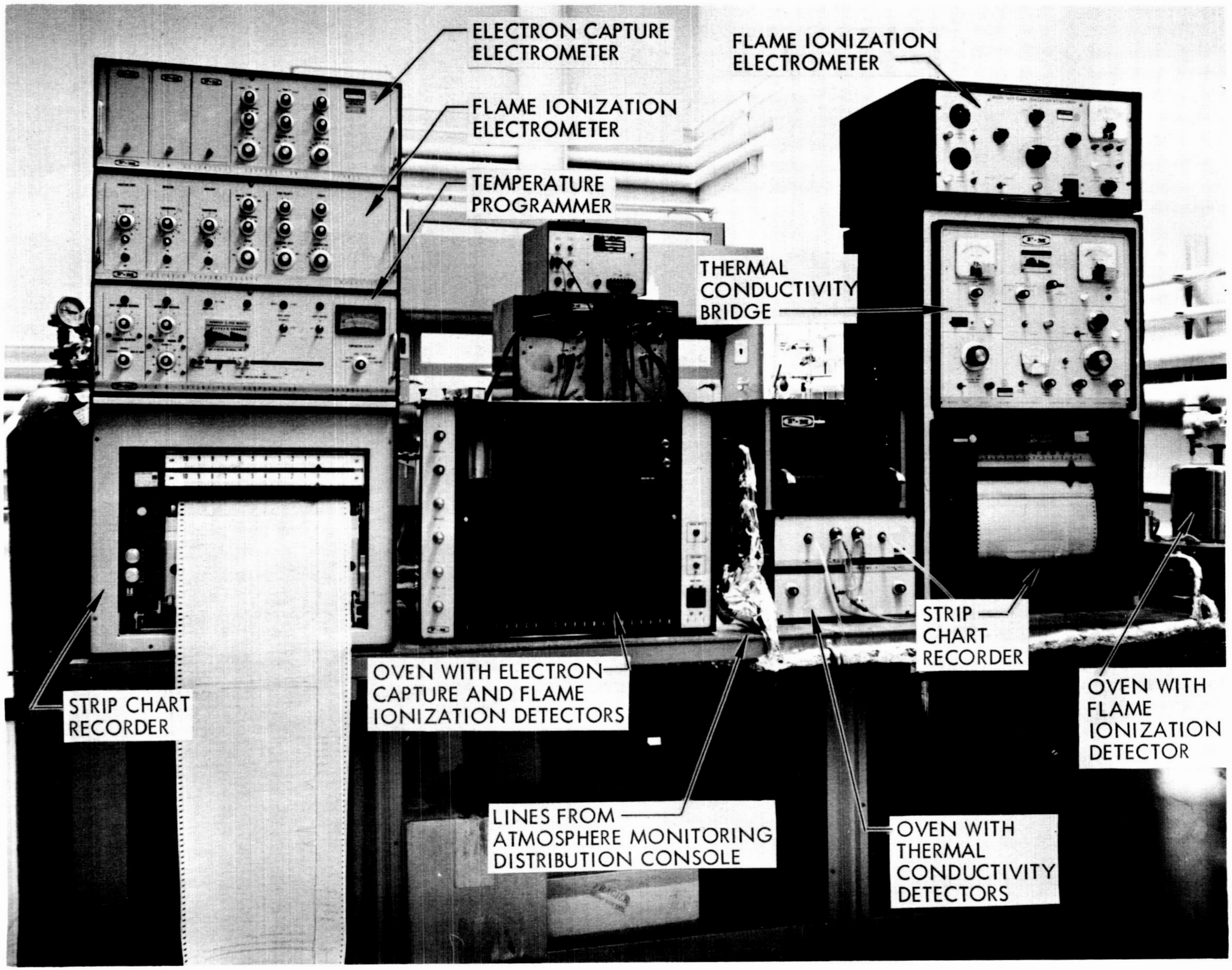

Figure 5-7 Gas Chromatographs Used for Atmosphere Monitoring

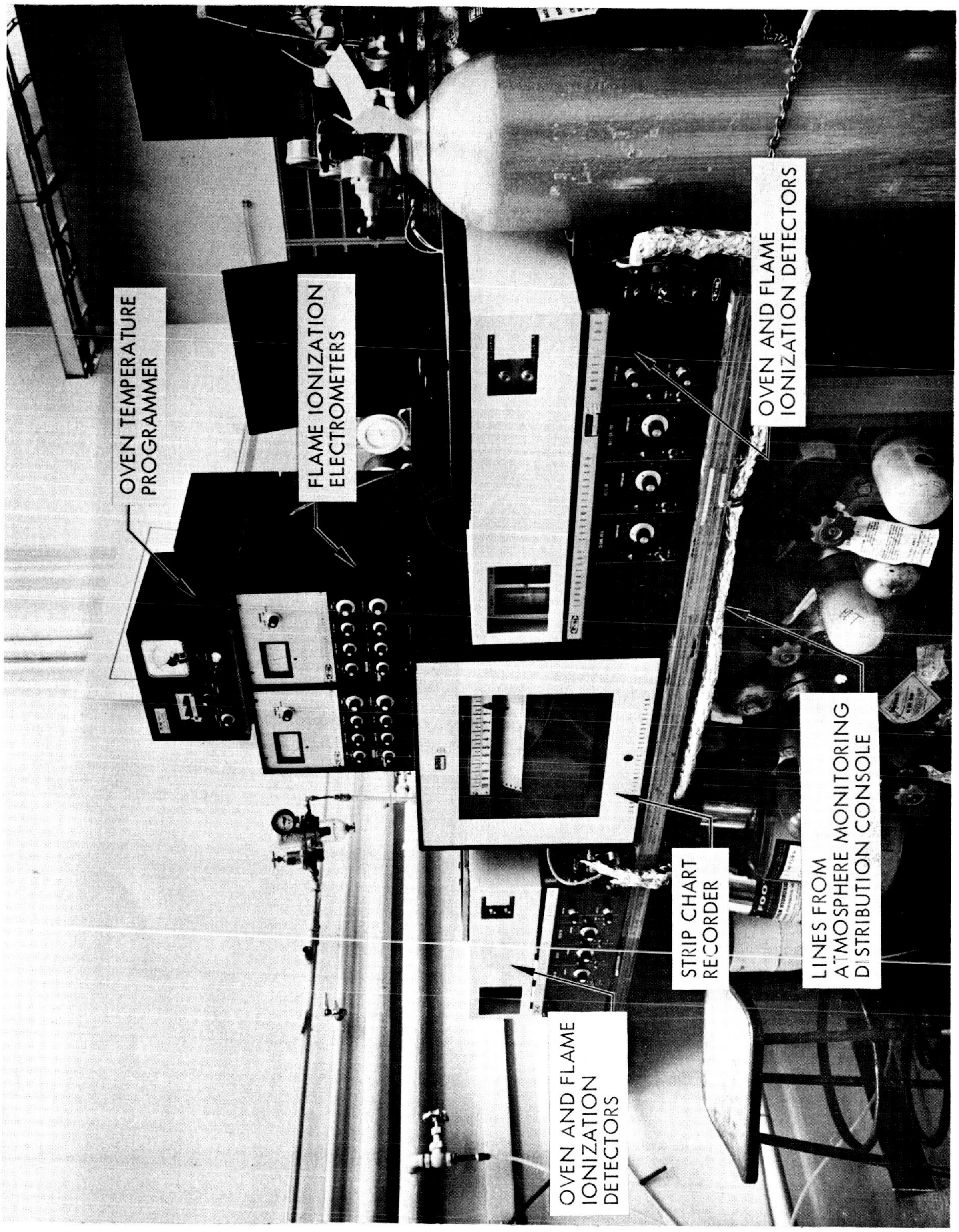

Figure 5-8 Additional Gas Chromatograph Used For Atmosphere Monitoring

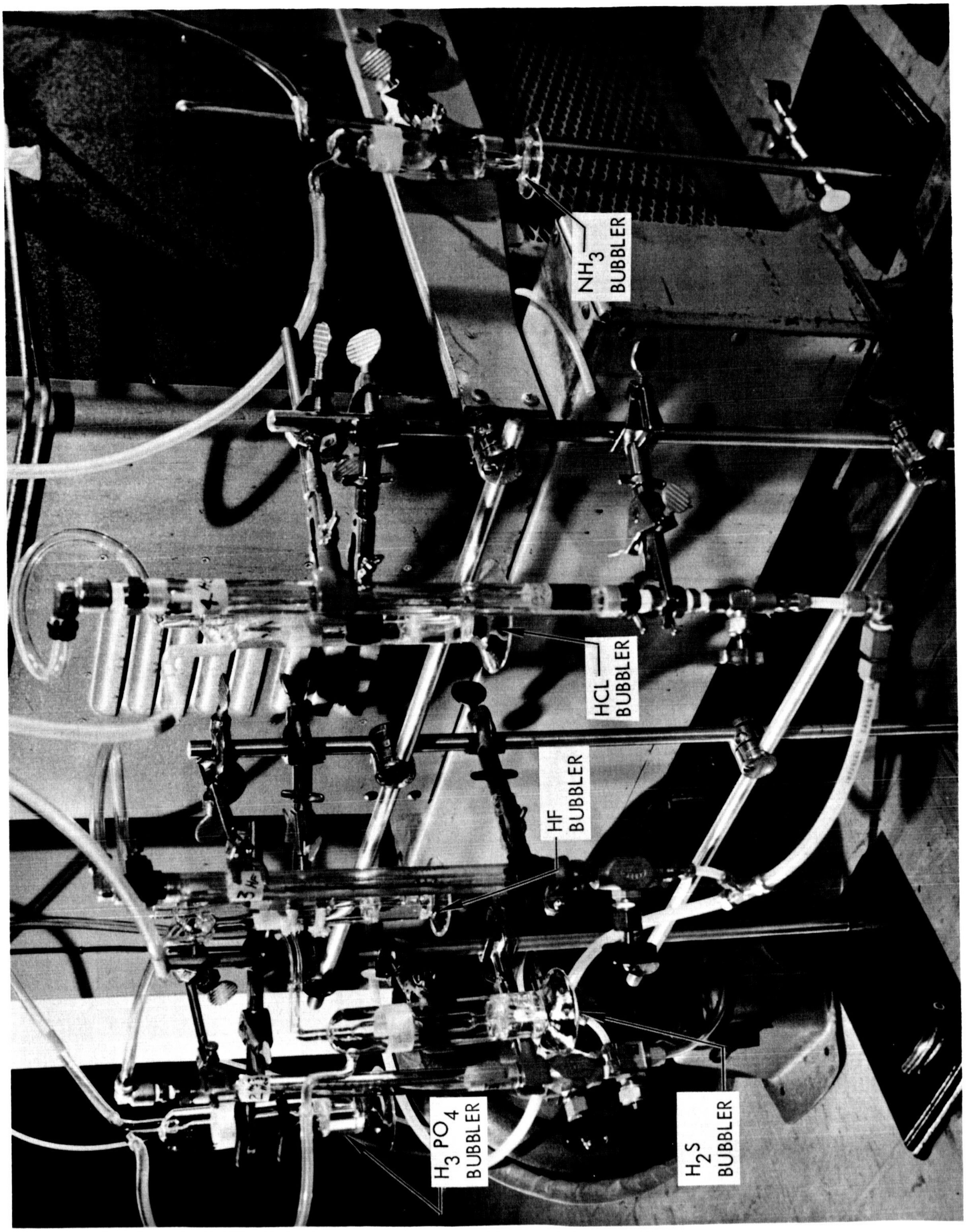

Figure 5-9 Bubblers For Colorimetric Analysis

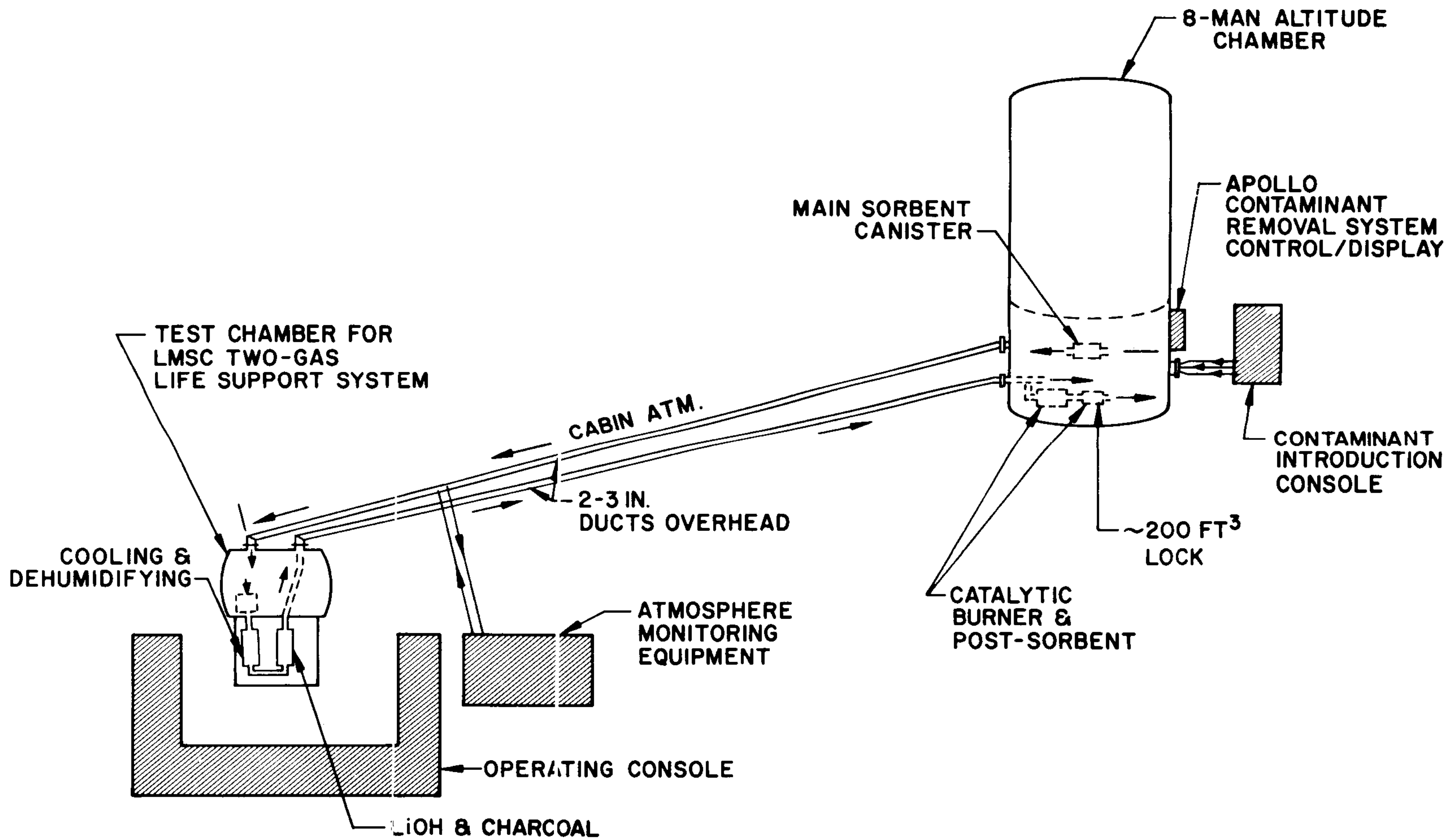

Figure 5-10 Test Area Layout For Closed-Chamber Evaluation

5.3 PROCEDURE

The contaminant removal unit, including catalytic oxidizer, post sorbent and main sorbent, was installed in the chamber lock as shown in Figure 5-11. The lock pressure was controlled to 5 psia, using a pure oxygen gas supply. On 16 August 1965 all systems were energized, and the contaminants listed in Table 5-1, plus carbon dioxide, water vapor and heat, were introduced into the chamber at specified rates (contaminants totaling approximately 6.5 grams/day, excluding methane and hydrogen). Gas was withdrawn from the chamber at approximately 0.8 m^3/hr to allow for gas analysis and to maintain a high oxygen concentration.* Oxygen was admitted through a total pressure regulator to replace the 0.8 m^3/hr withdrawn. The system was then operated, and the contaminants introduced, for five days. During this time, data were taken on contaminant, oxygen and CO_2 concentrations; humidity; temperatures; pressure; flow rates; power consumption; and catalytic oxidizer-post sorbent pressure drop.

After five days of operation under normal conditions, quantities of Freon-12 and n-Butane sufficient to cause upset conditions were injected into the chamber. This was done with the contaminants listed in Table 5-1 also being introduced at normal rates. The concentrations of the two "upset" contaminants were monitored for eight hours. The test was terminated on 21 August 1965.

A second test was performed due to a catalytic oxidizer heater failure in the first test. On 2 September 1965 all systems were energized and contaminants were introduced for 15 hours. During this time data were taken on methane, hydrogen and carbon monoxide concentrations as well as on general system operating characteristics. After 15 hours under normal conditions, sufficient quantities of (1) carbon monoxide to cause an increase in concentration to 5 times SMAC and (2) methane and hydrogen to cause an increase in concentration to 2.5 times SMAC, were injected into the chamber. This was done with the contaminants listed in Table 5-1 being introduced at the specified rate. Concentrations of the upset contaminants were monitored for eight hours.

*Air leakage into the chamber from ambient provides a continuous nitrogen input.

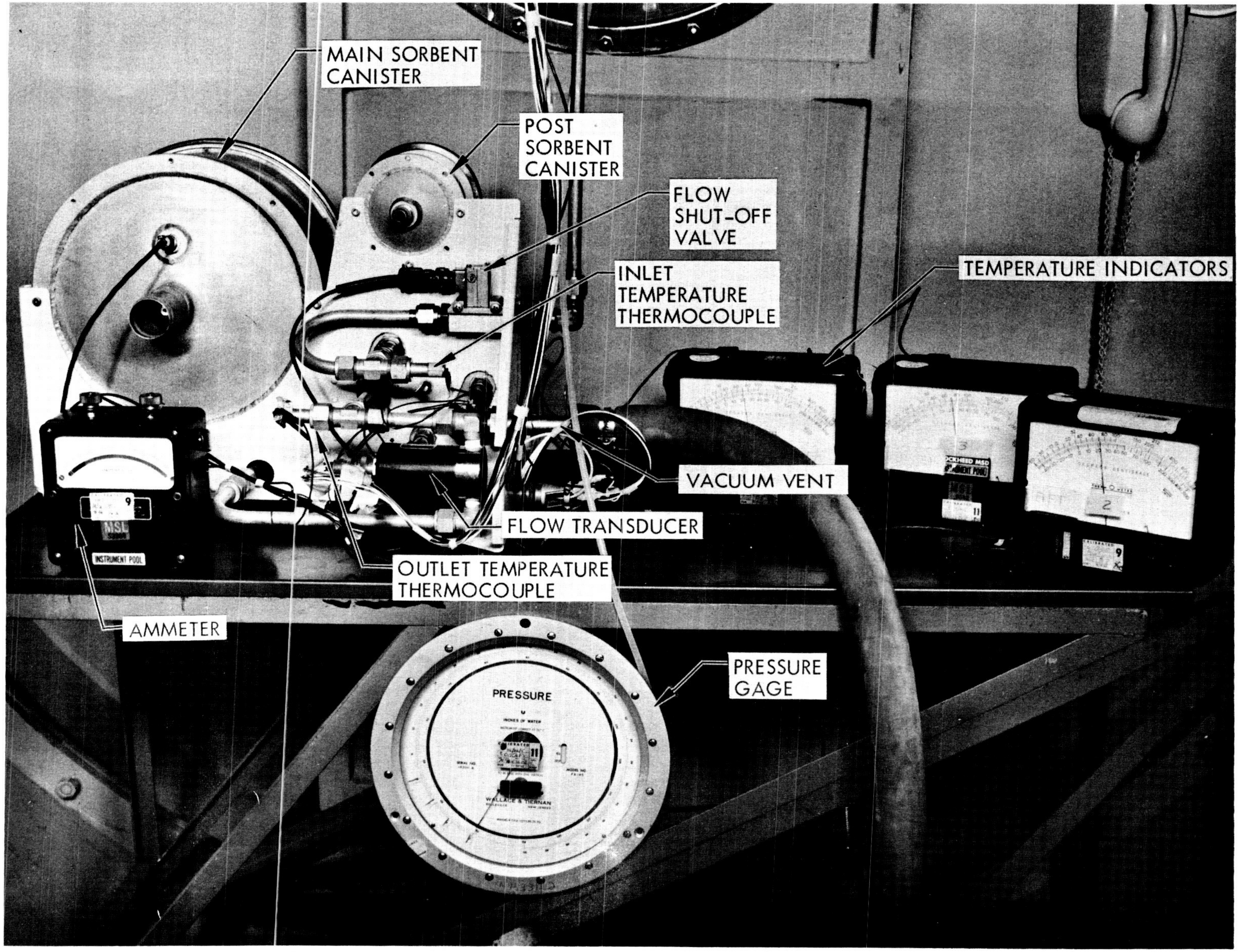

Figure 5-11 Apollo Contaminant Control System Installed in 8 Man Chamber Lock

Table 5-1

CONTAMINANTS INTRODUCED AND MONITORED
Closed Chamber Evaluation

CONTAMINANT	METHOD OF ANALYSIS(2) GC	COL.	KIT.	NET INTRO-DUCTION RATE (g/day)	SMAC (mg/m^3)	ESTIMATED INSTRUMENT SENSITIVITY (mg/m^3)	CONCENTRA-TION AFTER 5 DAYS (mg/m^3)
Freon 12	X			0.044	130.0	0.3	
1, 4-Dioxane	X			0.026	72.0	20.0	
Ammonia		X		4.6	7.0	1.5	11.5
Butene-1	X			0.012	60.4	0.3	0.3
trans-Butene-2	X			0.021	60.4	0.3	0.3
Ethyl Alcohol	X			0.026	50.0	10.0	
n-Butyl Alcohol	X			0.026	60.0	37.0	
Freon-11	X			0.069	147.0	0.3	
Freon-22	X			0.020	93.5	0.3	
Acetone	X			0.026	63.0	30.0	
Ethyl Acetate	X			0.026	92.0	58.0	
n-Hexane	X			0.047	94.6	10.0	
Trichloroethylene	X			0.047	104.0	40.0	
Toluene	X			0.047	99.0	18.0	
Freon-21	X			0.042	110.0	0.3	
n-Butane	X			0.010	26.5	0.3	0.3
Propyl Mercaptan	X			0.026	82.0	50.0	
Methyl Chloroform	X			0.047	143.0	25.0	
Carbon Monoxide	X			1.0	22.0	6.9	43.0(5)/30.0(4)
Acetylene	X			0.025	28.0	0.9	2.1(5)
Formaldehyde		X		0.019	1.0	0.5	0.4
Methyl Mercaptan	X			0.075	8.0	0.5	
Propylene	X			0.042	45.2	0.2	1.7
Methane	X			17.0	1720.0	17.0	790.0(5)
Hydrogen	X			1.1	215.0	3.6	76.0(5)
Freon-114	X			0.46	184.0	0.3	
Hydrogen Sulfide		X		0.033	6.0	0.05	0.05(3)
Monomethyl Hydrazine	X			0.047	49.6	10.0	
Nitrogen Tetroxide		X		0.012	1.8	0.02	0.08(3)
Sulfur Dioxide(1)			X	0.0	2.6	0.15	0.9
Phosphoric Acid(1)		X		0.0	0.2	0.09	0.04
Hydrogen Fluoride(1)		X		0.0	0.4	0.04	
Phosgene(1)			X	0.0	0.8	0.1	0.1
Hydrochloric Acid(1)		X		0.0	1.4	0.2	

(1) Contaminants monitored but not introduced
(2) GC – Gas Chromatography
Col. – Wet chemical colorimetry
Kit. – Kittagawa tubes
(3) Maximum during test
(4) Equilibrium value measured during retest of catalytic oxidizer
(5) Concentration at time of heater failure

5.4 RESULTS

Data on system total pressure, relative humidity, oxygen concentration and carbon dioxide concentration throughout the five-day test are shown on Figure 5-12. Contaminant concentrations during the five-day test for normal conditions are shown for each contaminant in alphabetical order in Figures 5-13 through 5-27. Data taken during the upset condition following the five-day test are shown on Figures 5-28 and 5-29. Data taken during the retest on 2 September are shown on Figures 5-30 through 5-32.

5.5 DISCUSSION

5.5.1 Five-Day Test

Desired Contaminant Introduction Rates. The desired contaminant introduction rates were based upon achieving a total contaminant introduction of 6.5 grams per day (excluding hydrogen and methane). Ammonia and carbon monoxide were to be introduced at the design removal rates. The remainder of the 6.5 grams per day was to be distributed evenly among the rest of the contaminants.

Hydrogen and methane were to be introduced at a total of 1.1 gm-moles/day. These contaminant introduction rates are shown in Table 5-2 under "Desired Net Introduction."

Introduction Rate Adjustment for Outflow Leakage. Under equilibrium conditions, the contaminants introduced into the chamber are removed by the contaminant removal system and by outflow leakage; i.e.,

$$\dot{m}_r = \dot{m}_t - \dot{m}_\ell \qquad (5.1)$$

where

$\dot{m}_r$ = rate of contaminant removal by system

$\dot{m}_t$ = total introduction rate

$\dot{m}_\ell$ = contaminant leakage rate

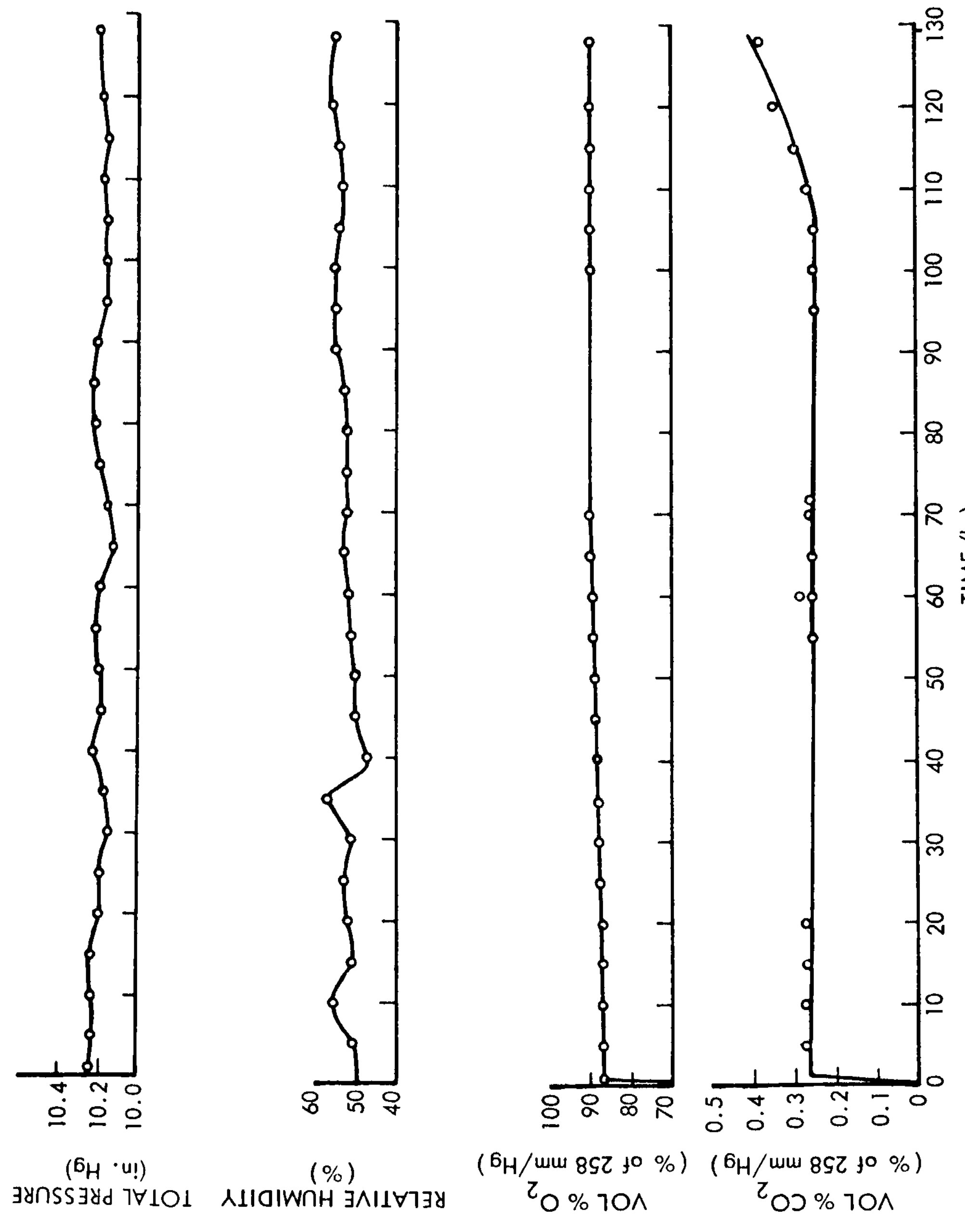

Figure 5-12 System Total Pressure, Relative Humidity, and Oxygen and CarbonDioxide Concentrations Throughout the Five-Day Test

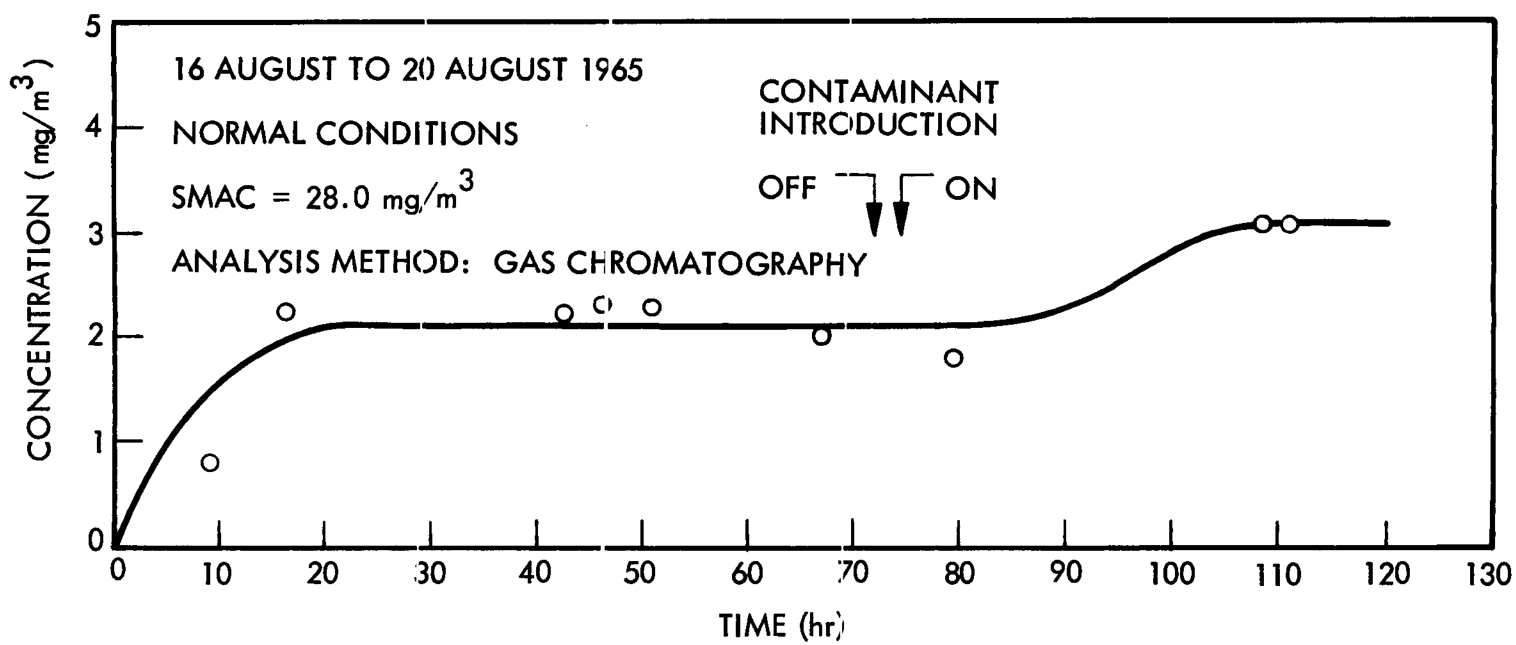

Figure 5-13 Acetylene Concentration

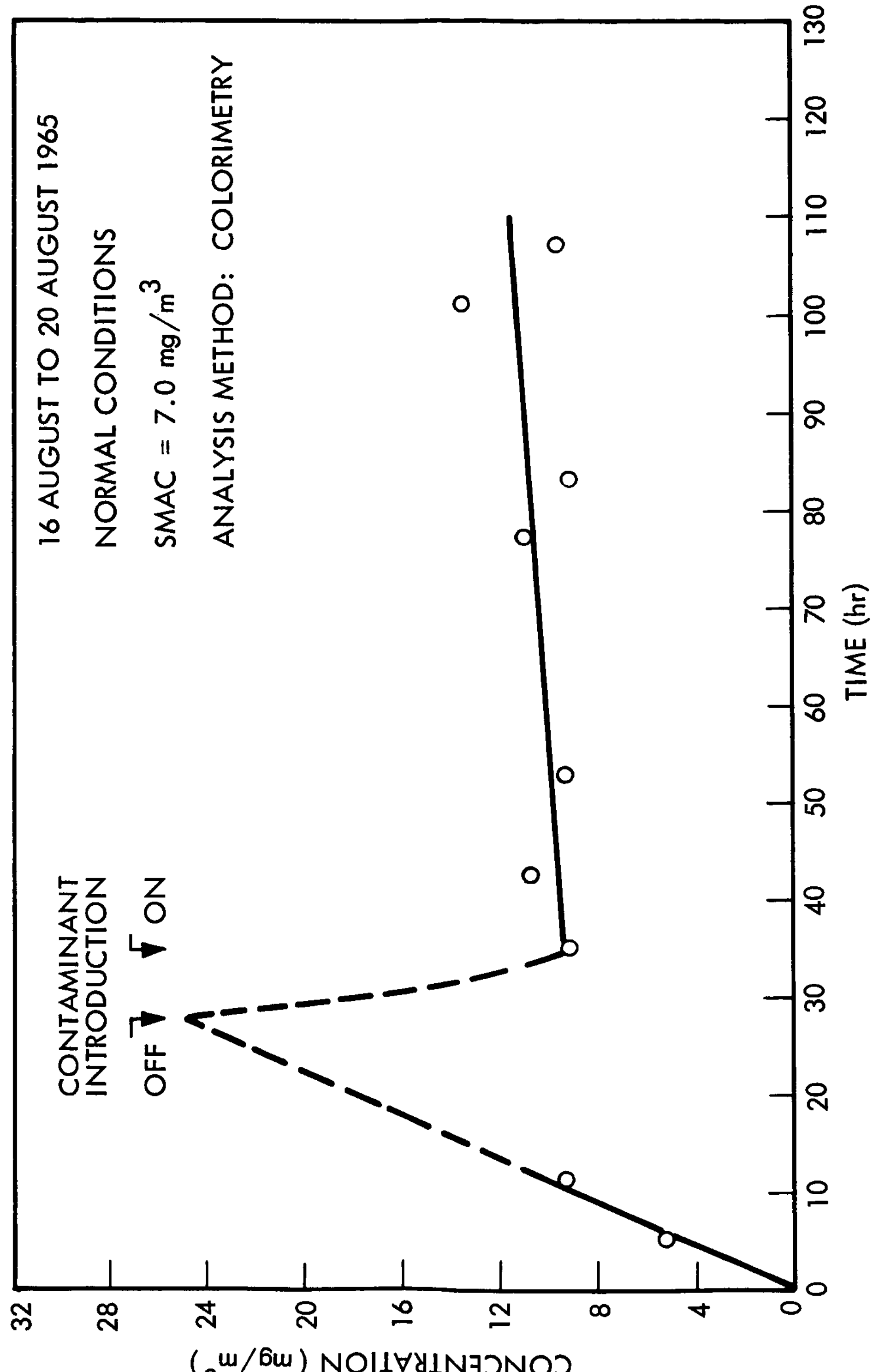

Figure 5-14 Ammonia Concentration

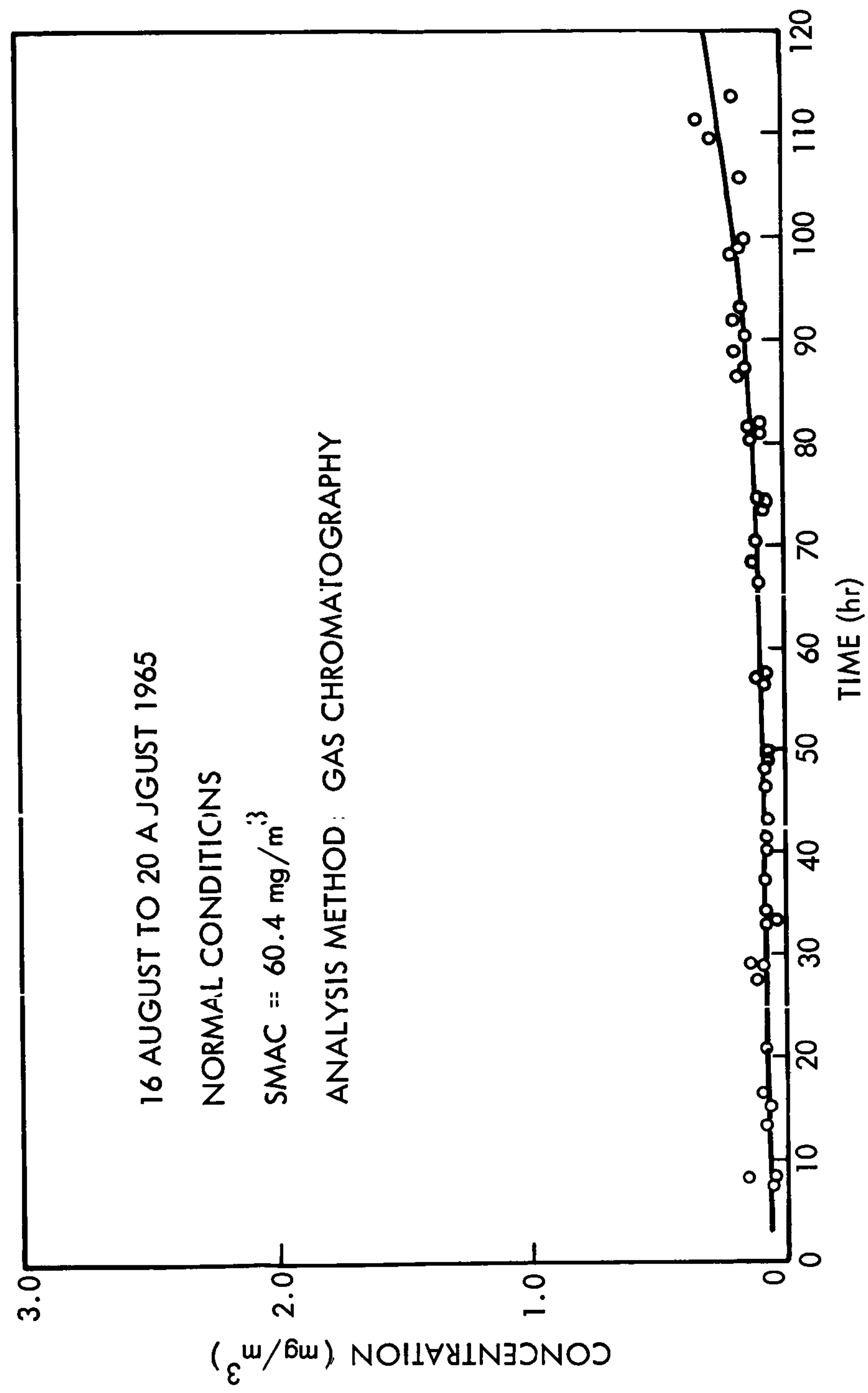

Figure 5-15 n-Butane Concentration

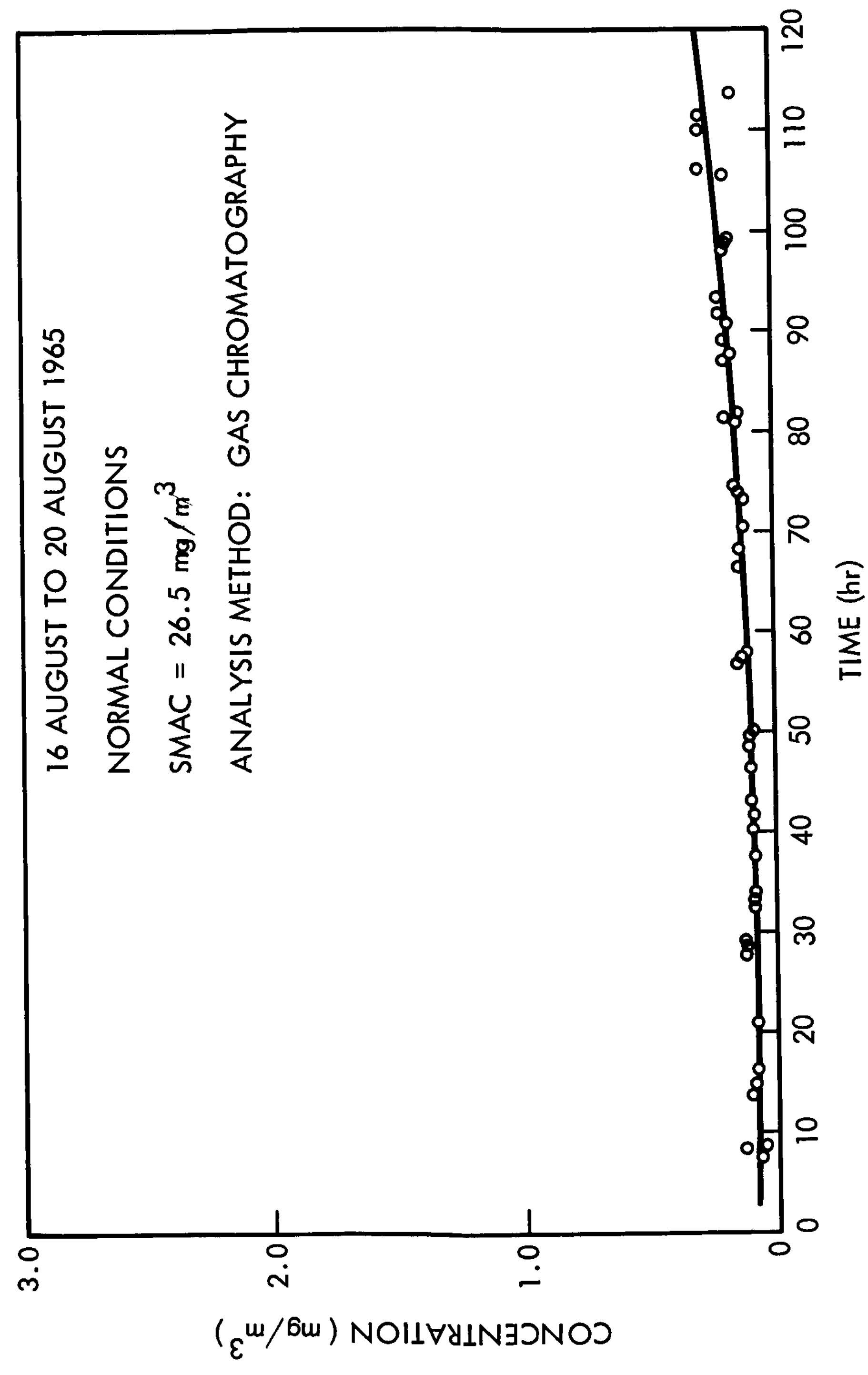

Figure 5-16 Butene-1 Concentration

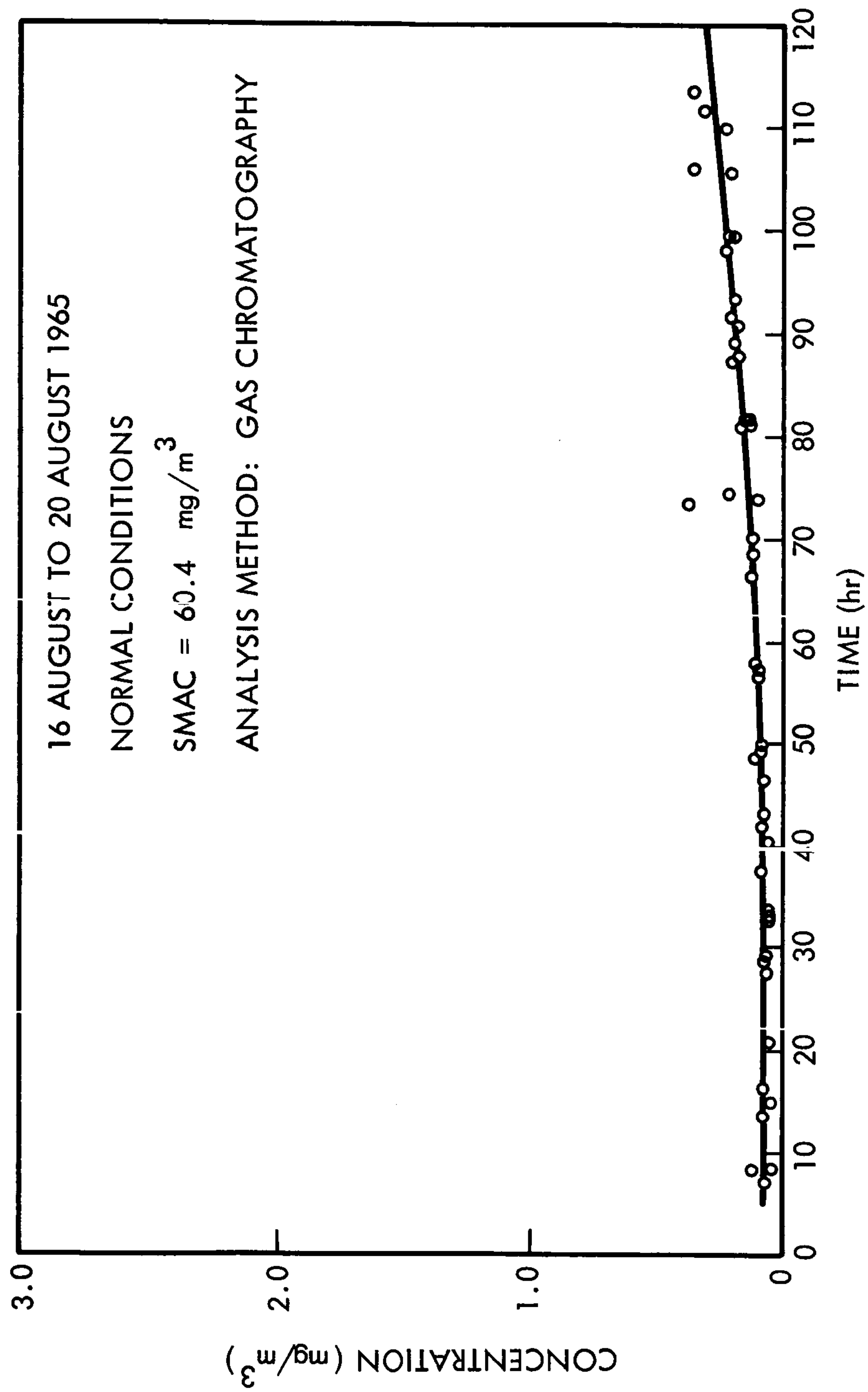

Figure 5-17 trans-Butene-2 Concentration

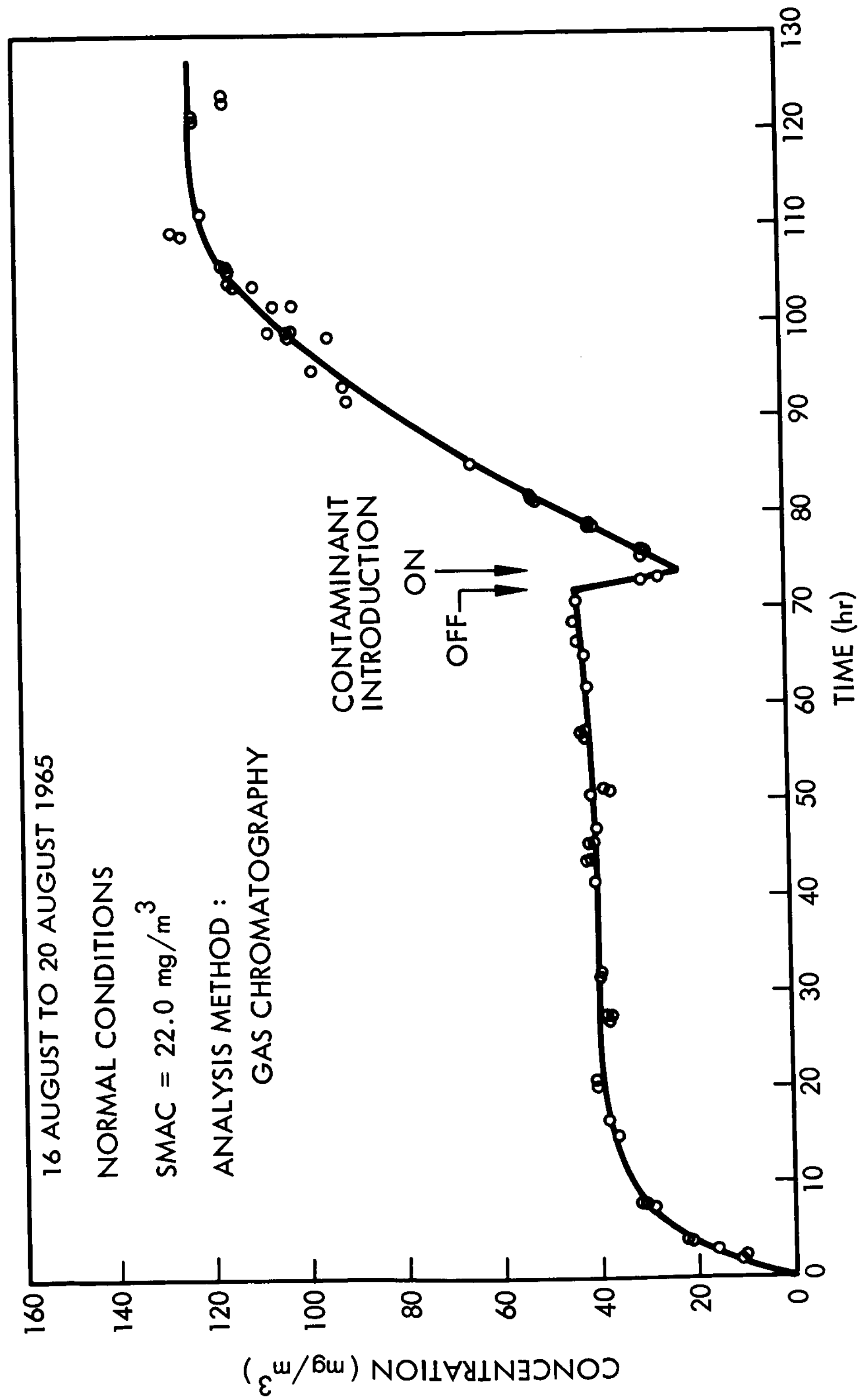

Figure 5-18 Carbon Monoxide Concentration

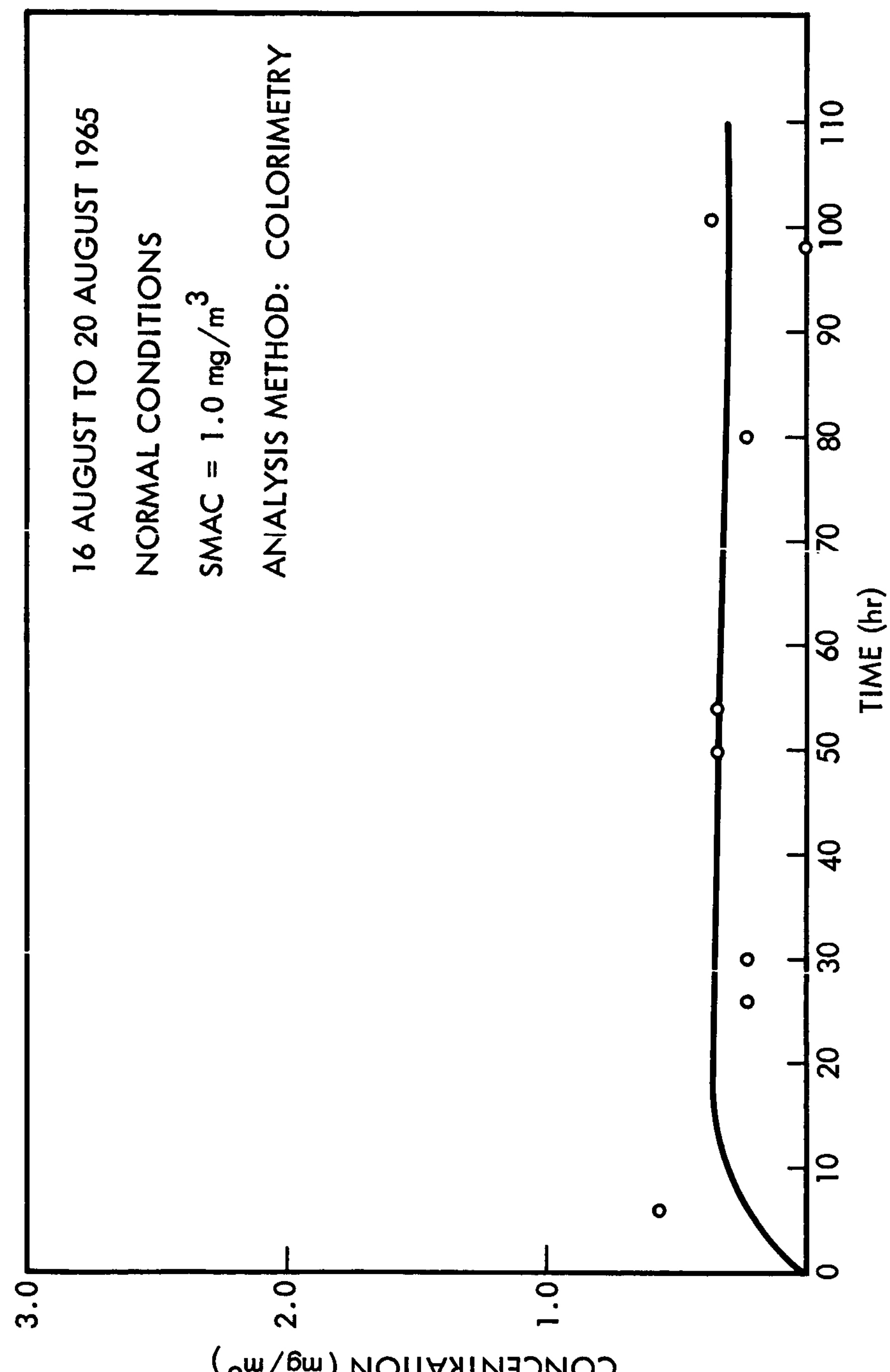

Figure 5-19 Formaldehyde Concentration

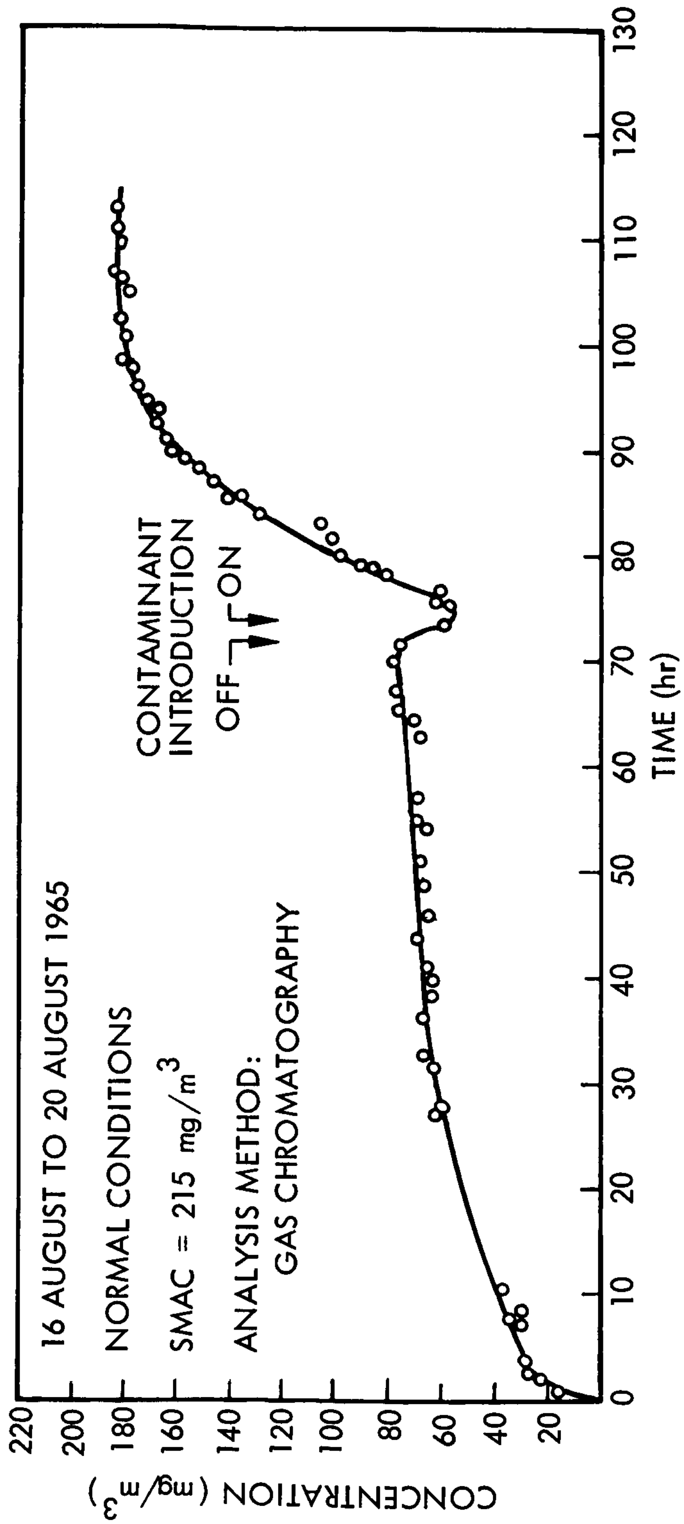

Figure 5-20 Hydrogen Concentration

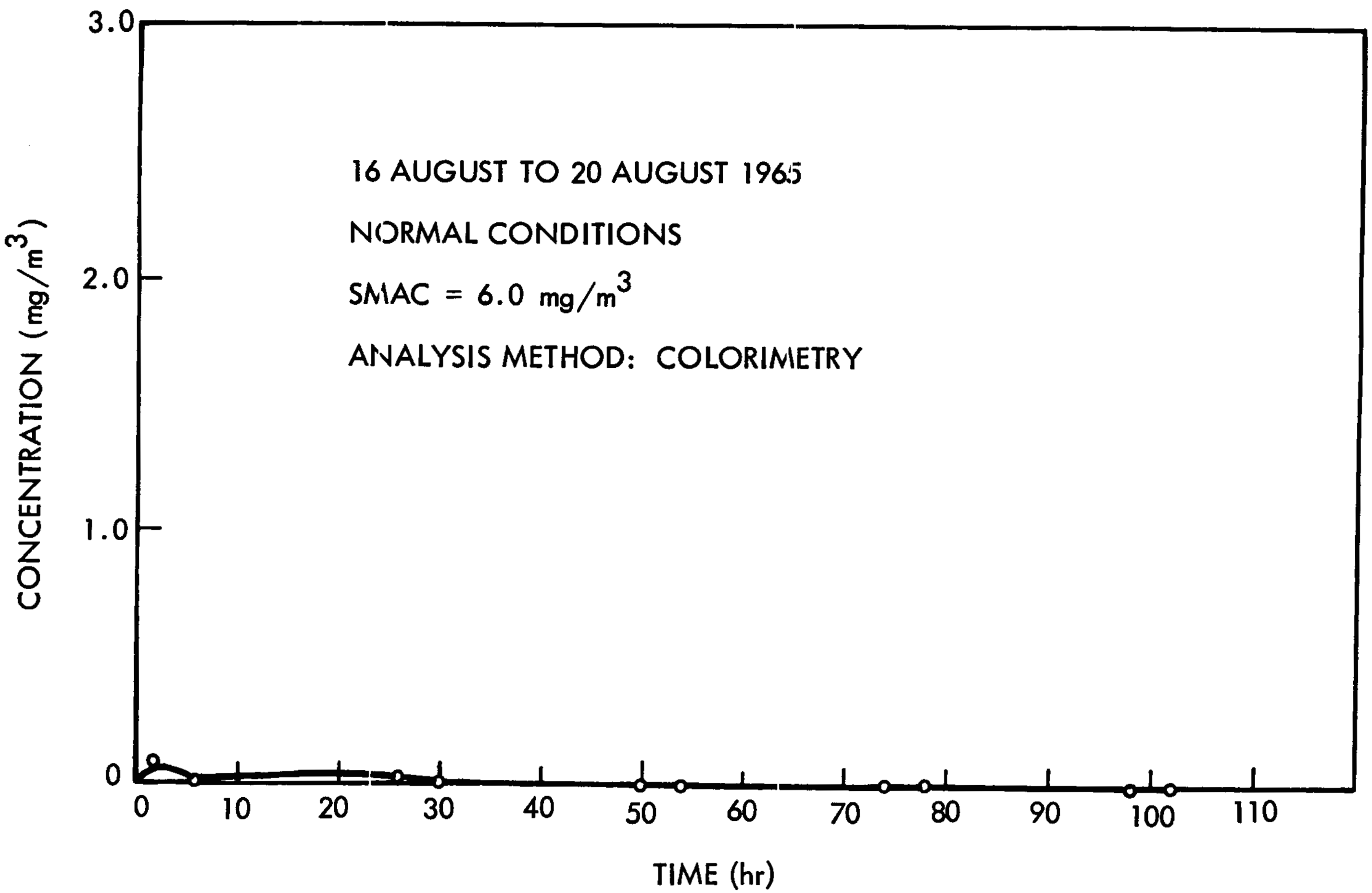

Figure 5-21 Hydrogen Sulfide Concentration

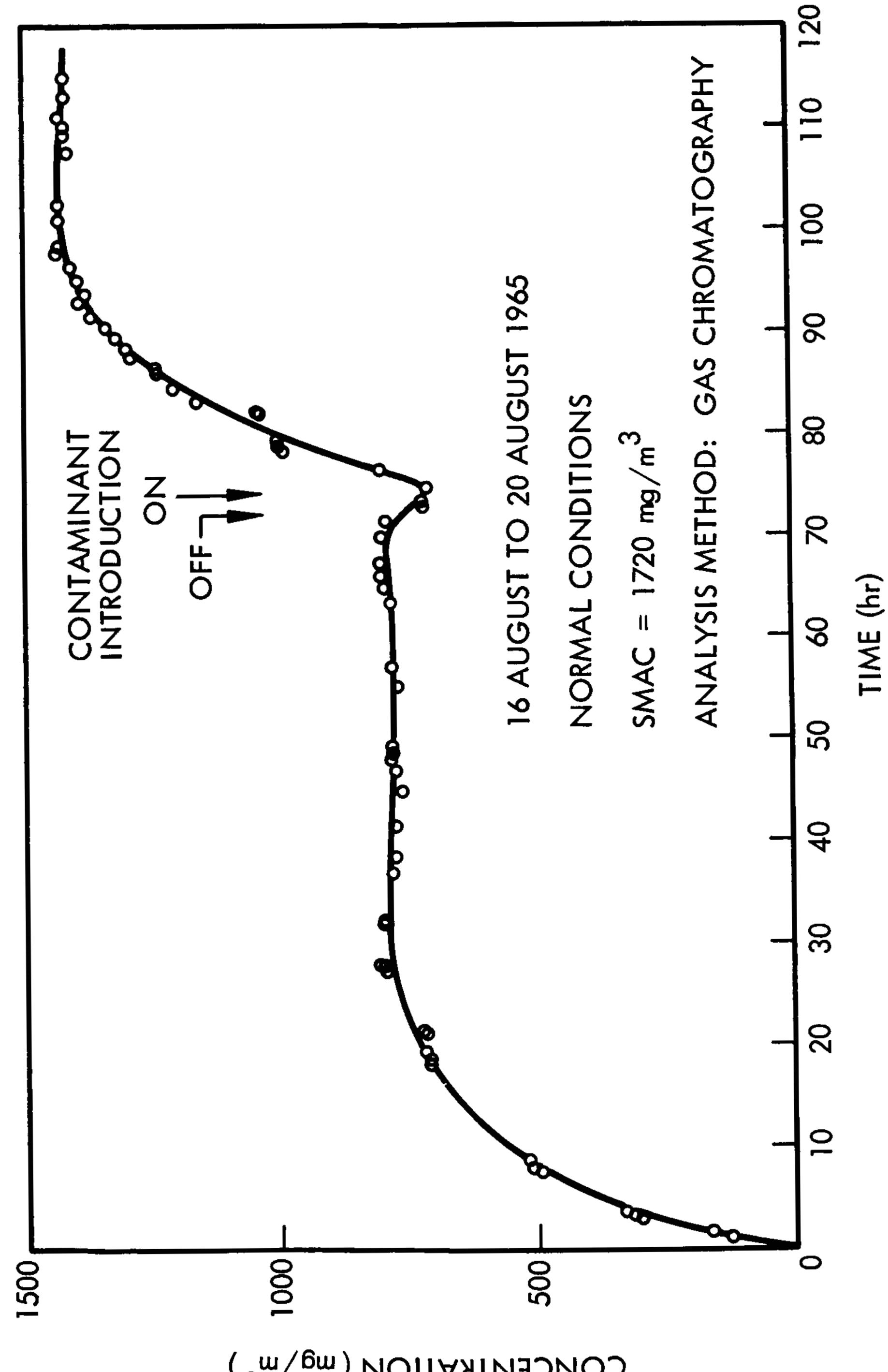

Figure 5-22 Methane Concentration

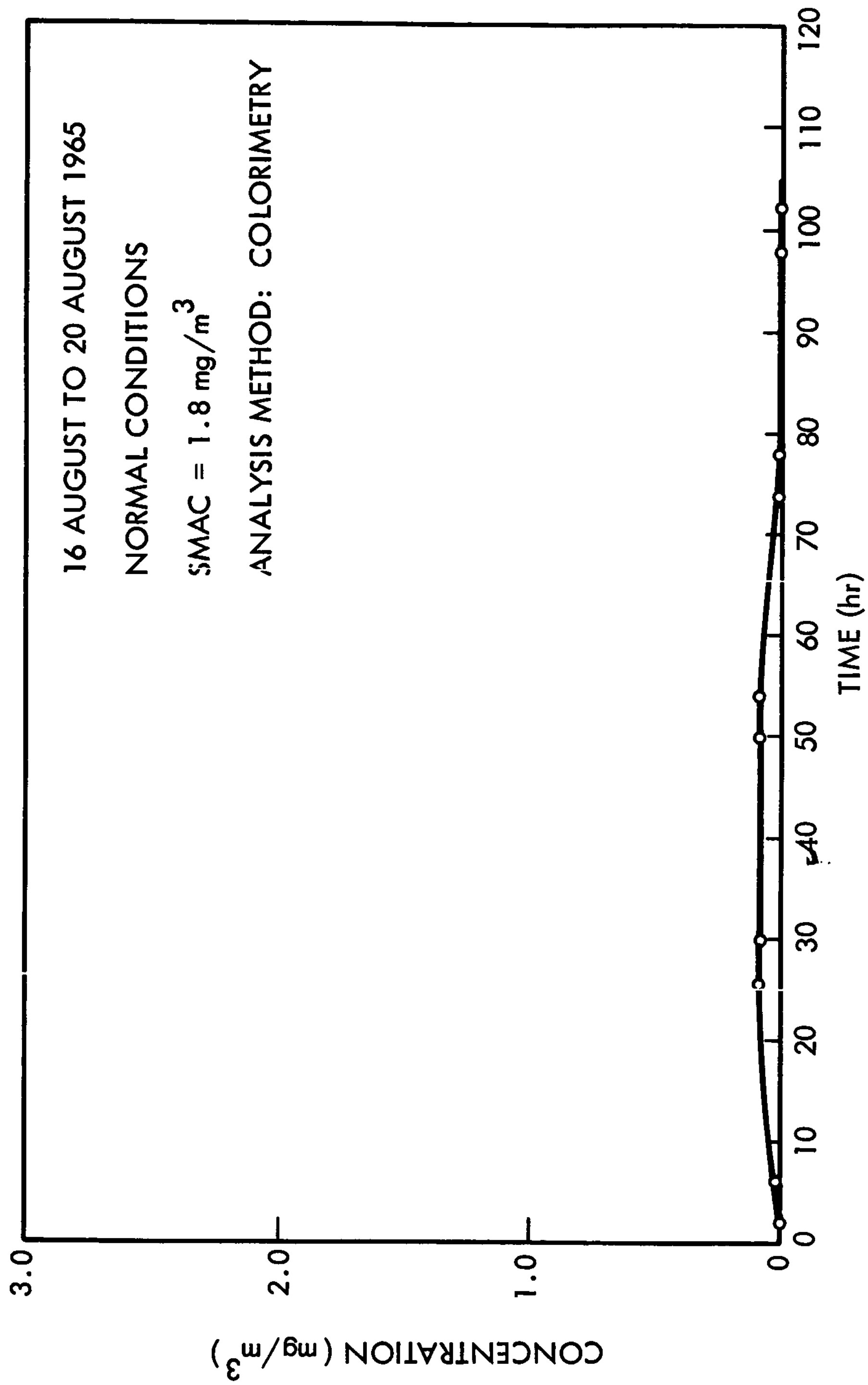

Figure 5-23 Nitrogen Dioxide Concentration

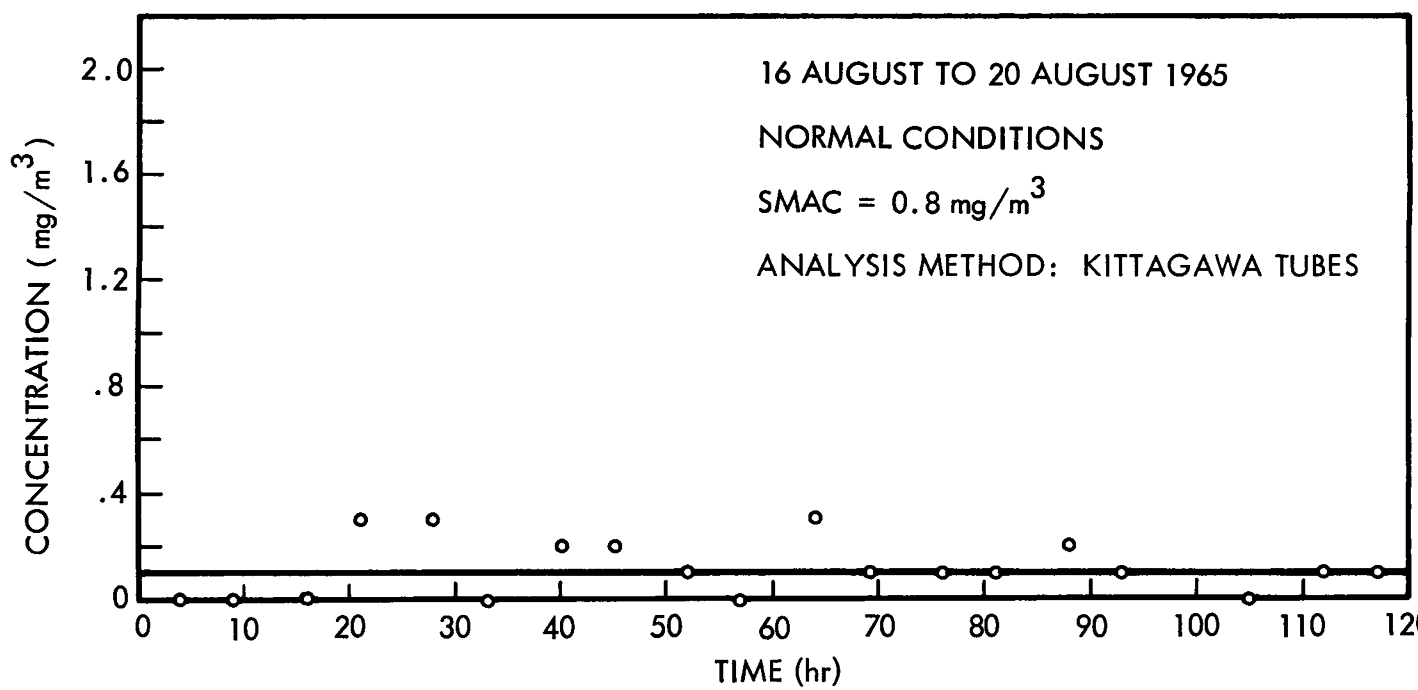

Figure 5-24 Phosgene Concentration

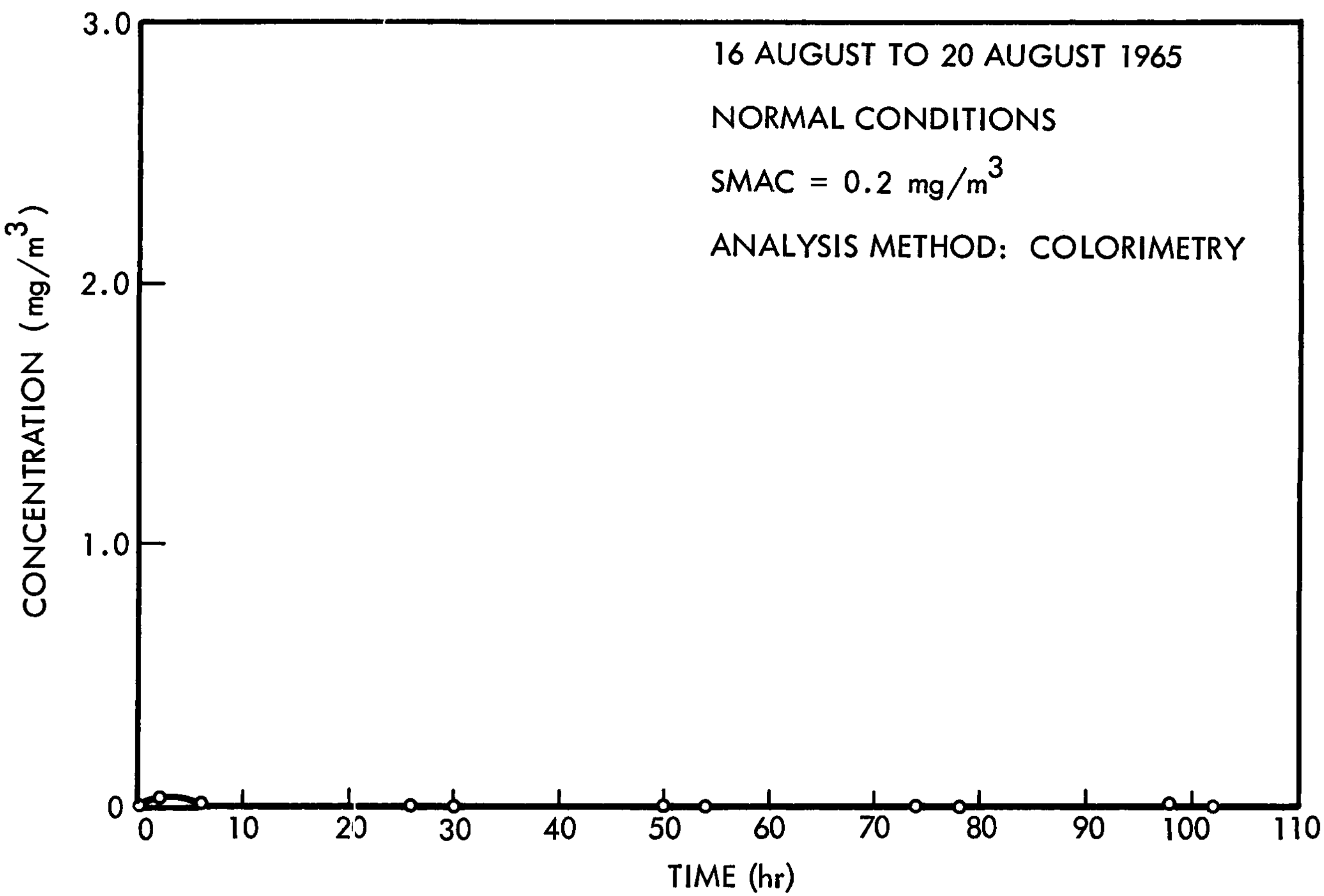

Figure 5-25 Phosphoric Acid Concentration

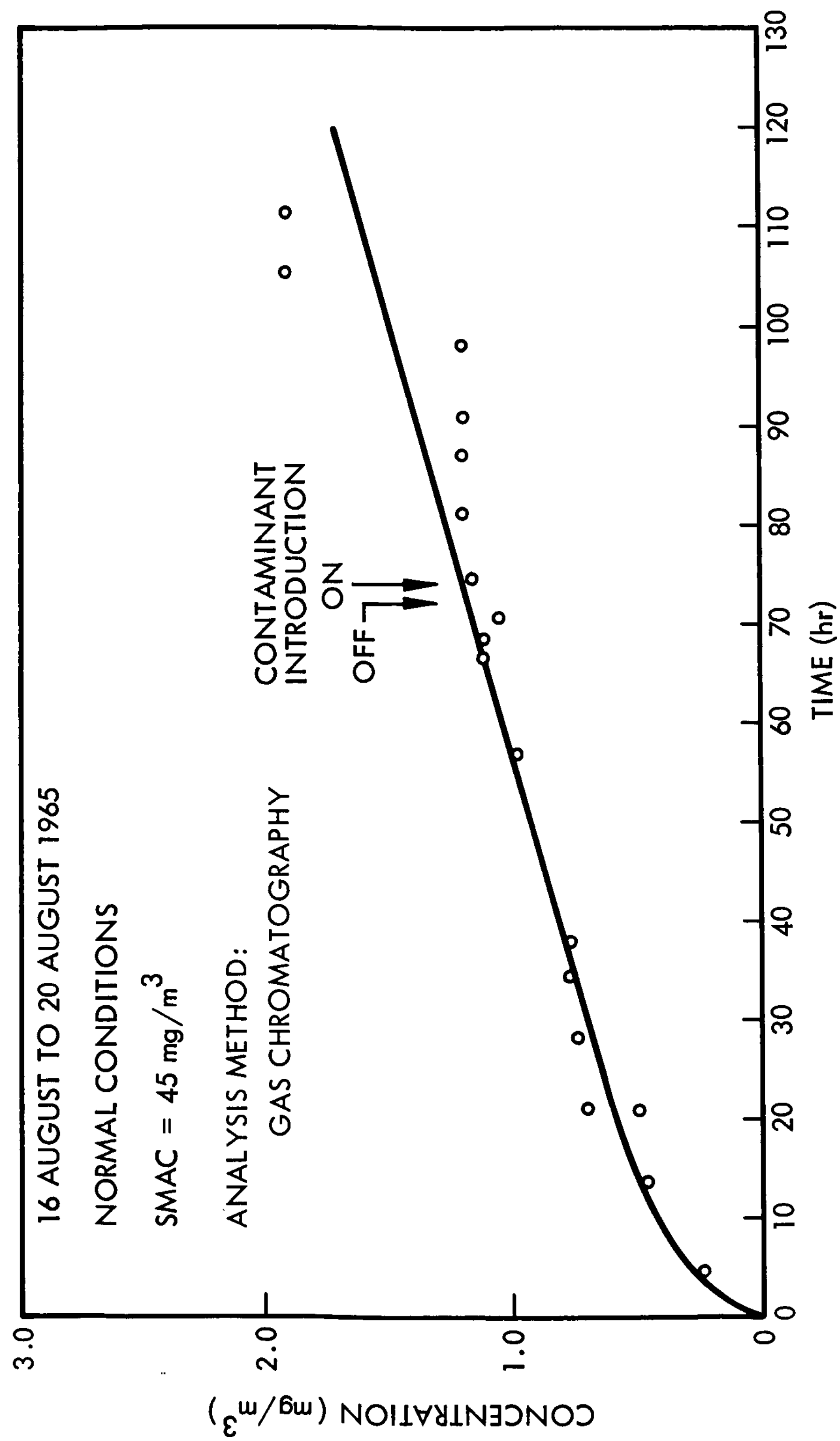

Figure 5-26 Propylene Concentration

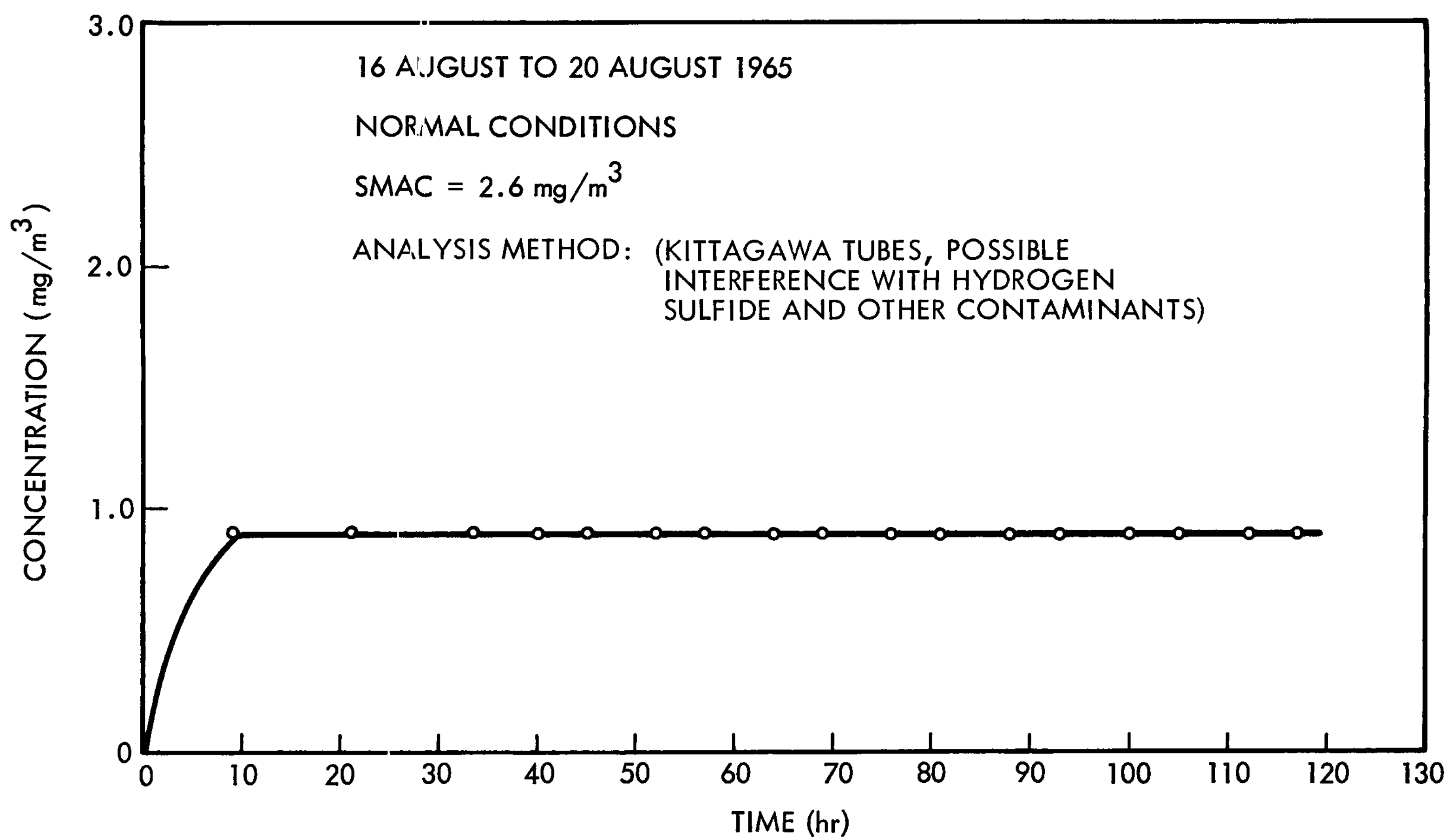

Figure 5-27 Sulfur Dioxide Concentration

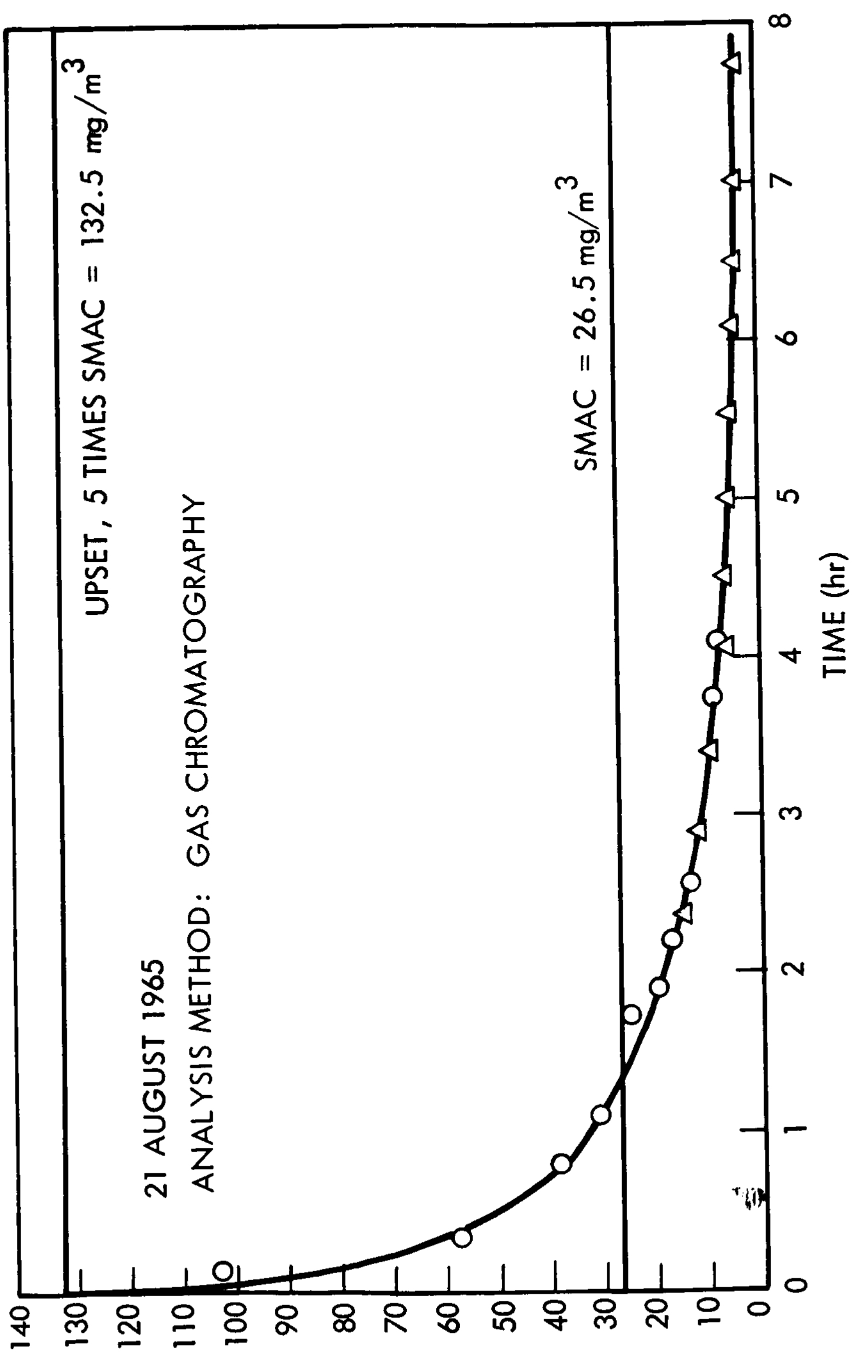

Figure 5-28 n-Butane Concentration, Upset Condition

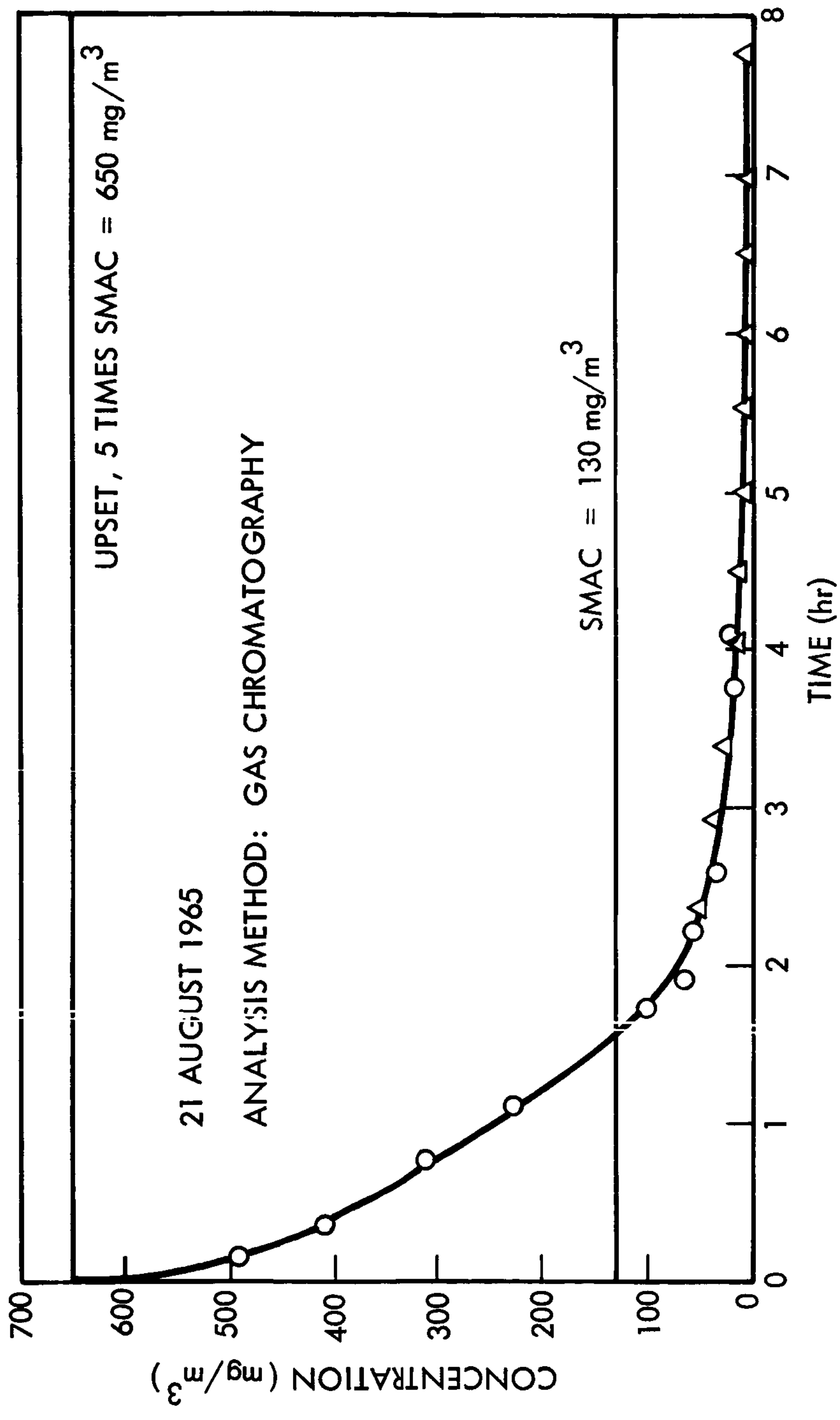

Figure 5-29 Freon 12 Concentration, Upset Concition

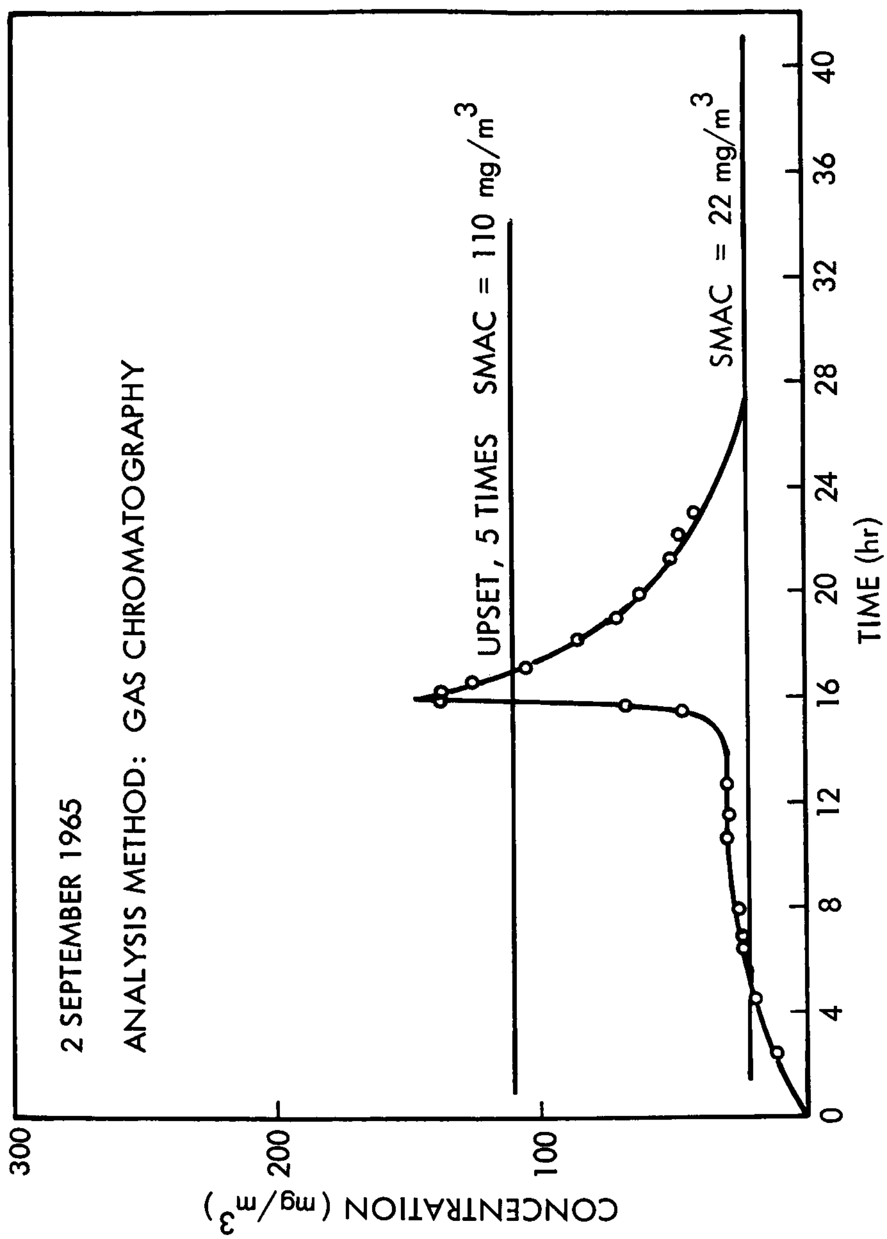

Figure 5-30 Carbon Monoxide Concentration, Upset Condition

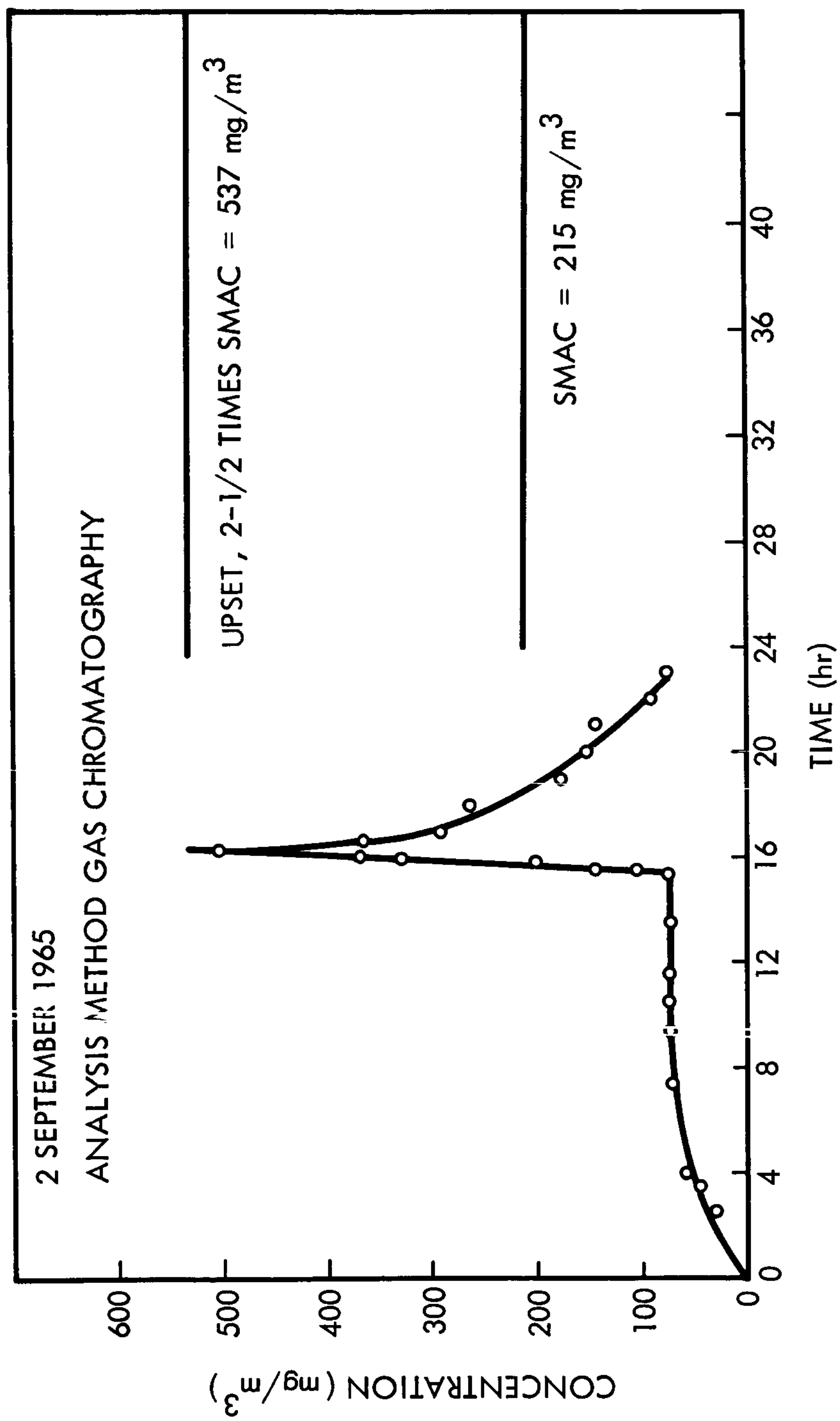

Figure 5-31 Hydrogen Concentration, Upset Condition

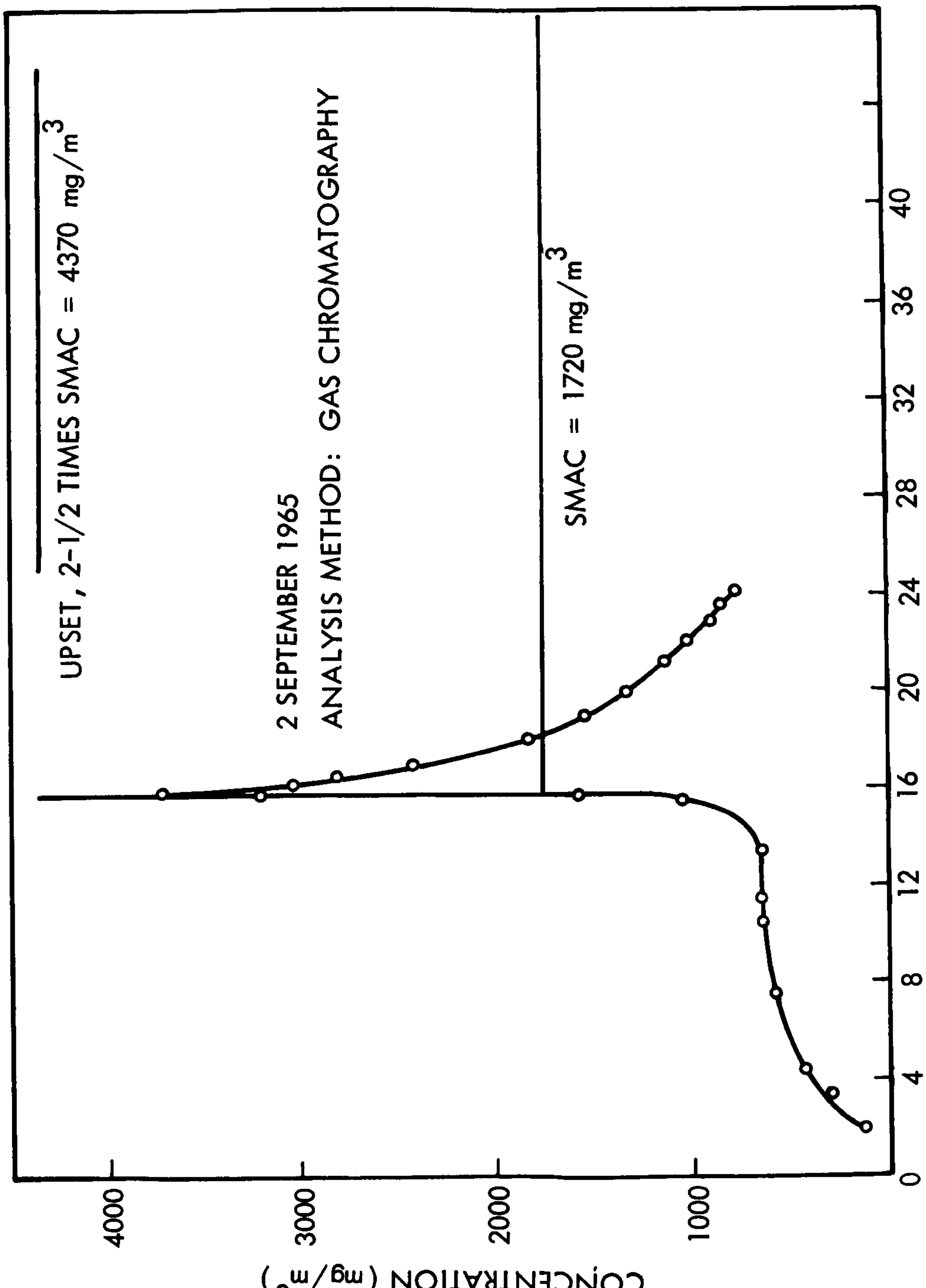

Figure 5-32 Methane Concentration, Upset Condition

Table 5-2

CONTAMINANT NET AND GROSS INTRODUCTION RATES

CONTAMINANT	DESIRED NET INTRODUCTION RATE (g/day)	DESIRED GROSS INTRODUCTION RATE (g/day)	ACTUAL GROSS INTRODUCTION (g/day)
Freon-12	0.0327	0.0327	0.0437
1, 4-Dioxane	0.0327	0.0327	0.0260
Ammonia	4.75	4.88	4.80
Butene-1	0.0327	0.0327	0.0184
trans-Butene-2	0.0327	0.0327	0.0265
Ethyl Alcohol	0.0327	0.0327	0.0260
n-Butyl Alcohol	0.0327	0.0327	0.0260
Freon-11	0.0327	0.0327	0.0690
Freon-22	0.0327	0.0345	0.0198
Acetone	0.0327	0.0327	0.0260
Ethyl Acetate	0.0327	0.0327	0.0260
n-Hexane	0.0327	0.0327	0.0470
Trichloroethylene	0.0327	0.0327	0.0470
Toluene	0.0327	0.0327	0.0470
Freon-21	0.0327	0.0327	0.0420
n-Butane	0.0327	0.0327	0.0159
Propyl Mercaptan	0.0327	0.0327	0.0260
Methyl Chloroform	0.0327	0.0327	0.0470
Carbon Monoxide	0.90	1.41	1.640
Acetylene	0.0327	0.051	0.077
Formaldehyde	0.0327	0.051	0.0260
Methyl Mercaptan	0.0327	0.051	0.0750
Propylene	0.0327	0.050	0.0730
Methane	8.8	28.0	28.0
Hydrogen	1.1	1.71	1.71
Freon-114	0.0327	0.0327	0.460
Hydrogen Sulfide	0.0327	0.0327	0.0327
Monomethyl Hydrazine	0.0327	0.0327	0.0470
Nitrogen Tetroxide	0.0327	0.0327	0.012
Sulfur Dioxide	0.0327	0.0327	0.0

Also

$$\dot{m}_\ell = F_\ell C \tag{5.2}$$

where

F_ℓ = outflow leakage

C = contaminant concentration

and

$$\dot{m}_r = F_r \eta_r C \tag{5.3}$$

where

F_r = flow rate through removal device

η_r = average removal efficiency

Combining equations 5.1, 5.2 and 5.3

$$\dot{m}_t = \dot{m}_r \left(1 + \frac{F_\ell}{F_r \eta_r}\right) \tag{5.4}$$

Consequently, the net introduction rate must be increased by the factor $(1 + F_\ell / F_r \eta_r)$ to account for removal by outflow leakage.

To develop this factor, it is necessary to know $F_r \eta_r$ for each of the contaminants introduced. Data from the Phase I effort were used for this purpose. Application of this factor resulted in the "Desired Gross Introduction Rate" values shown in Table 5-2.

Actual Introduction Rates. When the desired gross introduction rates were established, the contaminants were purchased in pre-mixed groups, selected by grouping non-reactive contaminants which had similar vapor pressures. The relative concentrations of contaminants within a group were selected to provide the relative desired gross contaminant introduction rates.

Actual gas concentrations were measured upon delivery to LMSC and found to deviate from requested concentrations. In view of this, input flow rates were adjusted to insure that the desired gross introduction of critical contaminants were maintained. Consequently, other contaminants were introduced at rates slightly in excess of the desired rates. The actual gross introduction rates are shown in Table 5-2, resulting in the net introduction rates shown in Table 5-1.

The difference between the gross and actual introduction rates is a function of $F_r \eta_r$ and F_ℓ. Since $F_r \eta_r$ varied from contaminant to contaminant, the differences between gross and actual introduction rates also vary. A notable example is methane. In this case the desired and actual gross introduction rates were identical but the desired and actual net introduction rates are different by almost a factor of 2.

The net contaminant introduction rates shown in Table 5-1 were established at the outset of the test. After 28 hours of testing, it was discovered that ammonia was being introduced at 11.5 g/day instead of the desired 4.75 g/day. This caused the ammonia concentration to exceed SMAC. At this point ammonia introduction was discontinued. The ammonia was then left off until its concentration returned to near SMAC, at which time (after 35 hours of testing) ammonia contaminant introduction at 4.75 grams/day was reinstated.

Contaminant introduction then continued normally until 72 hours of testing had elapsed. At this time the catalytic oxidizer heater failed and for reasons of safety the contaminants primarily removed by the catalytic oxidizer (Table 5-3) were turned off.

After determining that continued introduction of methane and hydrogen would not result in explosive concentrations, these contaminants were again introduced at the original rates, starting at the 74th hour. Contaminant introduction then continued unchanged for the remainder of the five day test.

Contaminant Concentrations. The contaminant concentration data taken during the five day test are shown in Figures 5-13 through 5-27. The method of analysis is indicated on the appropriate figure and in Table 5-1. Table 5-1 also indicates the estimated instrumentation sensitivity. It appears from examination of the data scatter in Figures 5-13 through 5-27 that in most cases the data acquisition accuracy was within the estimated sensitivity. For those contaminants not reported in Figures 5-13 through 5-27 the concentrations during the five day test were below the estimated instrument sensitivity shown in Table 5-1.

Comparison with Predicted Results – Catalytic Oxidizer. For the contaminants removed primarily by the catalytic oxidizer an estimate of the oxidizer removal efficiency is shown in Table 5-3. This table indicates the design removal efficiency, and the efficiencies obtained in the bench test and the final five day test.

The removal efficiencies obtained for carbon monoxide and hydrogen in the five day test were essentially the same as those experienced during the bench test. The conversion efficiency for methyl merceptan during the five day test was not determinable; this contaminant was below the level of detection.

The removal efficiency obtained for methane was less than that experienced in the bench test, but greater than the design value. This is again attributed to variations in the catalyst bed temperature between the different tests.

Acetylene removal efficiency was lower than that experienced during the bench test. This is attributed to the extremely low concentration of acetylene present. The acetylene concentration at the inlet of the catalytic oxidizer during the five day test was lower than the concentration experienced at the catalytic oxidizer outlet during the bench test.

Removal efficiencies are not shown in Table 5-3 for propylene and formaldehyde. Propylene did not show an abrupt change in concentration when the catalytic oxidizer was inoperative, but a gradual increase throughout the five day test. This occurred because a significant portion of the propylene removal occurred by adsorption on charcoal. The quantity of propylene introduced during the five day test was 210 mg and the charcoal capacity for propylene at a concentration of 1.7 mg/m^3 was 116 mg.

The formaldehyde concentration did not change after the catalytic oxidizer became inoperative; it remained at about 0.4 mg/m^3. Thus, while formaldehyde did not appear to be removed by the catalytic oxidizer, removal by some mechanism must have occurred; otherwise the equilibrium concentration for formaldehyde would have been 1.7 mg/m^3.

Removal efficiencies for the five day test were determined from the following relationship:

$$\eta_r = \frac{F_\ell}{F_r}\left(\frac{C_e}{C} - 1\right) \tag{5.4}$$

where

F_ℓ = outflow leakage
F_r = flow through the catalytic oxidizer
C_e = equilibrium contaminant concentration, catalytic oxidizer inoperative
C = equilibrium contaminant concentration, catalytic oxidizer operative

Table 5-3

REMOVAL EFFICIENCY FOR CONTAMINANTS EXPECTED TO BE REMOVED BY THE CATALYTIC OXIDIZER

(Normal Contaminant Introduction)

Contaminant	η_r Design	η_r Bench Test	η_r Final Test
Carbon Monoxide	1.0	0.79	0.82
Acetylene	1.0	0.97	0.24
Methyl Mercaptan	1.0	≥0.89	*
Hydrogen	1.0	0.83	0.80
Methane	0.25	0.81	0.41
Propylene	1.0	0.95	**
Formaldehyde	1.0	~0.0	**

*Below detectable levels during test
**Significant removal by other means

The above relation was obtained by combining

$$\dot{m}_t = (F_r \eta_r + F_\ell)\, C \tag{5.5}$$

and

$$\dot{m}_t = F_\ell C_e \tag{5.6}$$

where $\dot{m}_t$ = total rate of contaminant introduction.

Equation 5.4 was used to determine η_r because its variables are known to a greater degree of accuracy than are those in equation 5.5.

For all the contaminants listed in Table 5-3, an estimate of F_ℓ, using equation 5.6, results in values between 0.6 m^3/hr and 1.0 m^3/hr. F_ℓ is a constant; an average value of 0.8 m^3/hr was assumed. Variations in F_ℓ for various contaminants are due to errors in either $\dot{m}_t$ or C_e. The solution of equation 5.4 does not require knowledge of $\dot{m}_t$ or the absolute value of C_e. The ratio C_e/C is more accurate than absolute values of C_e or C due to the elimination of possible calibration errors. The chromatograph detectors used have a linear response to concentration and thus a calibration error affects all concentrations equally.

The average outflow leakage, determined to be 0.8 m^3/hr during the test, had been estimated prior to the test at 0.9 m^3/hr (0.8 m^3/hr withdrawn through the atmosphere monitoring console and 0.1 m^3/hr leakage from the 8-man chamber lock to the main chamber). Since 0.8 m^3/hr was withdrawn from the system by the atmosphere monitoring console, it is concluded that the leakage from the lock to the main chamber was negligible during the final test. The two chambers were held within 0.4 psia of each other; consequently, the potential for leakage was very low.

Comparison With Predicted Results – Sorption Processes. Contaminants removed by sorption processes, whose concentrations and introduction rates were measured, are listed in Table 5-4. If it is assumed that removal efficiency, η_r , is not a function of concentration then the observed performance can be extrapolated to performance at SMAC. Column five of Table 5-4 lists the contaminant removal rates obtained by such an extrapolation.

It should be noted that the predicted values for removal rate in Table 5-4 are largely limited by saturation capacity rather than dynamic performance characteristics. If column five exceeds column six, the system has more than sufficient dynamic capability to fully utilize the sorbent capability. If column six exceeds column five, the opposite is true.

Table 5-4

CONTAMINANTS REMOVED BY SORPTION PROCESSES

Contaminant	SMAC (mg/m^3)	$C^{(1)}$ (mg/m^3)	$\frac{SMAC}{C}$	$\dot{m}_{intro}$ (g/day)	$\dot{m}_r^{(2)}$ (g/day)	$\dot{m}_r^{(3)}$ (g/day) predicted
Ammonia	7.0	11.5	0.61	4.6	2.8	4.75
n-Butane	60.4	0.3	201	0.01	2.0	5.04
Butene-1	26.5	0.3	88.4	0.012	1.1	3.09
trans Butene-2	60.4	0.3	201	0.012	4.2	3.96

(1) Test concentration after five days
(2) Extrapolated contaminant removal capability at SMAC, assuming η_r = constant
(3) Ref. 1, p. 7-24

Comparison of the last two columns of Table 5-4 indicates that at SMAC, ammonia would be removed at 2.8 g/day. Since the test concentration was close to SMAC, this extrapolation is probably valid. Because of the low concentration of the other contaminants measured, relative to SMAC, the other values in column five are probably unduly conservative.

Comparison with Predicted Results – Products of Oxidation. During the catalytic oxidizer bench test, no phosgene was observed to be created. Oxides of sulfur and nitrogen were generated but observed to be completely removed by the post-sorbent canister.

During the 5-day test, phosgene, sulfur dioxide, and nitrogen dioxide were detected in the chamber. This was not an unreasonable condition due to the larger quantities of halogens, and sulfur and nitrogen compounds in the chamber. After the catalytic oxidizer heater failed, however, the nitrogen dioxide level dropped below detectability, whereas the phosgene and sulfur dioxide levels remained unchanged.

Since phosgene and sulfur dioxide were being measured with Kittagawa tubes, the question of interference was raised. A check was made of all gaseous contaminant introduction bottles using phosgene and sulfur dioxide Kittagawa tubes. The gas mixture from the bottle containing CO, acetylene, propylene, methyl mercaptan and hydrogen produced a full-scale discoloration of the sulfur dioxide tube. A similar test of all gas bottles produced no indication on the phogene tube, however. Other contaminants introduced as liquids have not yet been checked.

As a result of the above investigation, the sulfur dioxide data are not meaningful. The phosgene tube will be checked further and results reported in the Phase III report.

If phosgene was indeed present in the chamber after the catalytic burner shutdown, it obviously was not an oxidation product from that device.

A recent investigation by Saunders (Ref. 5) in connection with contaminants detected in the MESA experiment, points out a potential sequence of events leading to phosgene, starting with trichloroethylene, which was being introduced. This sequence is (1) decomposition of trichloroethylene to dichloroacetylene due to contract with a strong base such as LiOH and (2) decomposition of the dichloroacetylene to phosgene and carbon monoxide.

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5. Naval Research Laboratory Letter Report, The Source and Identity of the Toxicant in the Project MESA Atmosphere, 6110-247A:RAS:vmg, 6 Oct 1965.

If phosgene was present in the chamber at the same level before and after the catalytic oxidizer shutdown, the test results would tend to support Saunder's hypothesis regarding the MESA experience. Additional testing with small concentrations of trichloroethylene at the inlet to a LiOH canister should clarify the situation.

5.5.2 Upset Conditions

For those contaminants removed primarily by sorption, the upset test was performed at the conclusion of the five day test. The upset test for those contaminants removed primarily by catalytic oxidation was conducted two weeks later. Separate tests were required due to a catalytic oxidizer heater failure during the five day test.

Contaminant Introduction. At the end of the five day test, 327 cc of n-Butane and 778 of Freon 12 were injected separately into the 200 ft^3 test chamber. This quantity of contaminant caused the concentrations of n-Butane and Freon 12 to rise to five times SMAC, as shown on Figures 5-28 and 5-29. Simultaneously, all of the contaminants listed in Table 5-1 were introduced at normal rates.

At the conclusion of the above test, the catalytic oxidizer heater was replaced and operation of all systems, under normal conditions, was started in the same manner as the five day test. Normal system operation was continued for approximately 16 hours at which time the upset contaminants were injected.

This upset consisted of injecting 580 cc of carbon monoxide, 68.5 liters of methane and 77.4 liters of hydrogen into the chamber. It can be seen from Figures 5-30, 5-31 and 5-32 that methane and hydrogen reached, and carbon monoxide exceeded, the upset value.

Comparison with Predicted Values. The contaminant concentrations four hours after each upset condition are shown in Table 5-5. This table includes the concentration measured during this test and the predicted concentration. Predictions were determined by equation 11, p. 7-26, Ref. 1, using three leakage conditions, the removal efficiency for each contaminant measured during the closed chamber test prior to the upset, the actual initial and upset concentrations, and the actual net introduction rates.

Table 5-5

CONTAMINANT LEVELS FOUR HOURS AFTER UPSET CONDITIONS

CONTAMINANT	SMAC (mg/m^3)	CONCENTRATION AFTER 4 HOURS (mg/m^3)			
		Actual	Predicted, Based on Measured Removal Efficiency		
			Test Chamber Leakage	Apollo Leakage (0.12 cfm)	Zero Leakage
n-Butane	26.5	7.5	29.0	46.0	51.5
Freon 12*	130.0	15	12.1	17.8	19.8
Carbon Monoxide	22.0	60	57	71	76
Methane	1720.0	1380	2130	2750	3030
Hydrogen	215.0	150	190	220	273

*Since Freon-12 was not observed during the operating period at normal conditions the measured removal efficiency was based on a concentration equal to the instrument sensitivity.

Since the test chamber leakage was greater than the assumed Apollo leakage of 0.12 cfm, the predicted test concentrations at four hours are correspondingly lower. The actual concentrations are, in two cases (CO and F-12) higher than the values predicted for the chamber test and in three cases (n-Butane, CH_4, and H_2) lower.

CO is about 5 percent high which can be considered well within the accuracy of prediction.

The Freon-12 prediction is subject to uncertainty due to the assumption of normal concentration equal to instrument sensitivity, but in any event is not in substantial disagreement with the predicted value.

The improved performance for the remaining contaminants can only be attributed to an increased removal efficiency resulting from the increased concentration experienced during the period of upset clearing. Table 5-4 shows that n-Butane was removed at less than the predicted rate at the low concentration achieved during the five-day test. The prediction in Table 5-4 was based on Phase I experiments with n-Butane near SMAC. Consequently, the effect noted during upset clearing bears out the effect of increasing η_r with C for n-Butane.

As shown in Table 4-2, during the bench-test upset η_r for hydrogen was increased by approximately 10 percent during upset conditions, whereas methane was not affected and carbon monoxide conversion increased only 5 percent. The methane and hydrogen concentrations reached during the chamber upset were higher than those achieved during the bench test (methane more than twice as high) further suggesting an increase in η_r with concentration.

In view of the test results one would expect that the concentrations in the last two columns of Table 5-5 would be reasonable estimates for CO and Freon-12, but higher than actual for the other three contaminants.

Upset clearing performance for CO, CH_4, and H_2 can be improved by increasing the flow through the catalytic oxidizer. Performance with Freon-12 is satisfactory with zero leakage; n-Butane is predicted to be in this category also when the actual and predicted test results are compared. These results generally agree with the predictions made in the Phase I report.

5.5.3 Heater Failure and Repair

The failure of the catalytic oxidizer heater was caused by a crack in the ceramic insulation which allowed the nichrome heater wire to completely oxidize when it contacted essentially pure oxygen at elevated temperature.

In constructing the replacement heater, the ceramic insulating material was cured in two steps, (1) a 24-hour room temperature air dry and (2) a 24-hour 200° F oven dry. The first heater had been cured for 24 hours at room temperature.

Section 6
INSTALLATION IN APOLLO COMMAND MODULE

6.1 INTEGRATION WITH APOLLO ECU

At a meeting between cognizant NASA, NAA-SID, and LMSC personnel, it was determined that the equipment arrangement shown in Figure 6-1 (shut-off valve-catalytic oxidizer-post sorbent assembly in parallel with the pressure suits) was considerably simpler than the arrangement presented in the Phase I report. The arrangement shown in Figure 6-1, which is recommended, is the simpler solution (in terms of plumbing) but will increase the power requirement slightly since the inlet temperature is approximately 50°F, rather than 130°F for the location recommended in the Phase I report. The only practical approach to providing 10 cfm for the main sorbent bed is the approach chosen, i.e., a self-contained fan in the main sorbent canister.

6.2 LOCATION OF HARDWARE

The most practical location for the contaminant removal unit was found to be an area presently designated for clothing storage. This area is just above the ECU and in close proximity to the suit supply and return ducts as shown in Figure 6-2. Drawings were obtained for this area in the Apollo Command Module and a half-size mockup made to determine feasibility of hardware installation. It was determined that the hardware could be placed in this area in a number of different arrangements. The arrangement shown in Figures 6-2 and 6-3 appeared to be the most desirable. In this arrangement the inlet and outlet to the shut-off valve-catalytic oxidizer-post sorbent assembly are easily interfaced with the suit supply and return ducts. As shown in Figure 6-4, the main sorbent canister fan discharges cabin atmosphere through the left-hand opening and draws cabin atmosphere in through the right-hand opening. The desired controls and displays can be located on the panel face as shown in Figure 6-4.

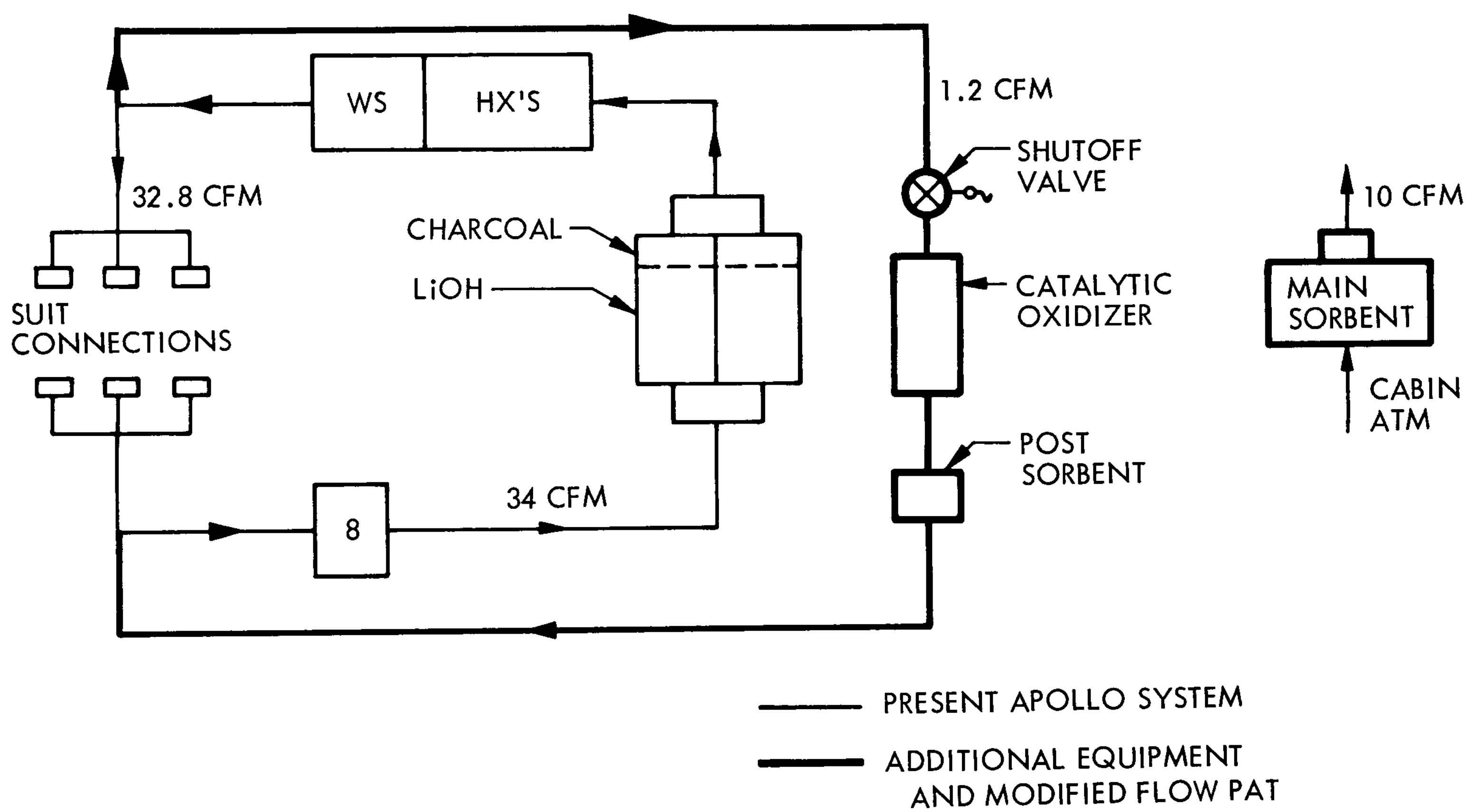

Figure 6-1 Interface With Apollo Environmental System

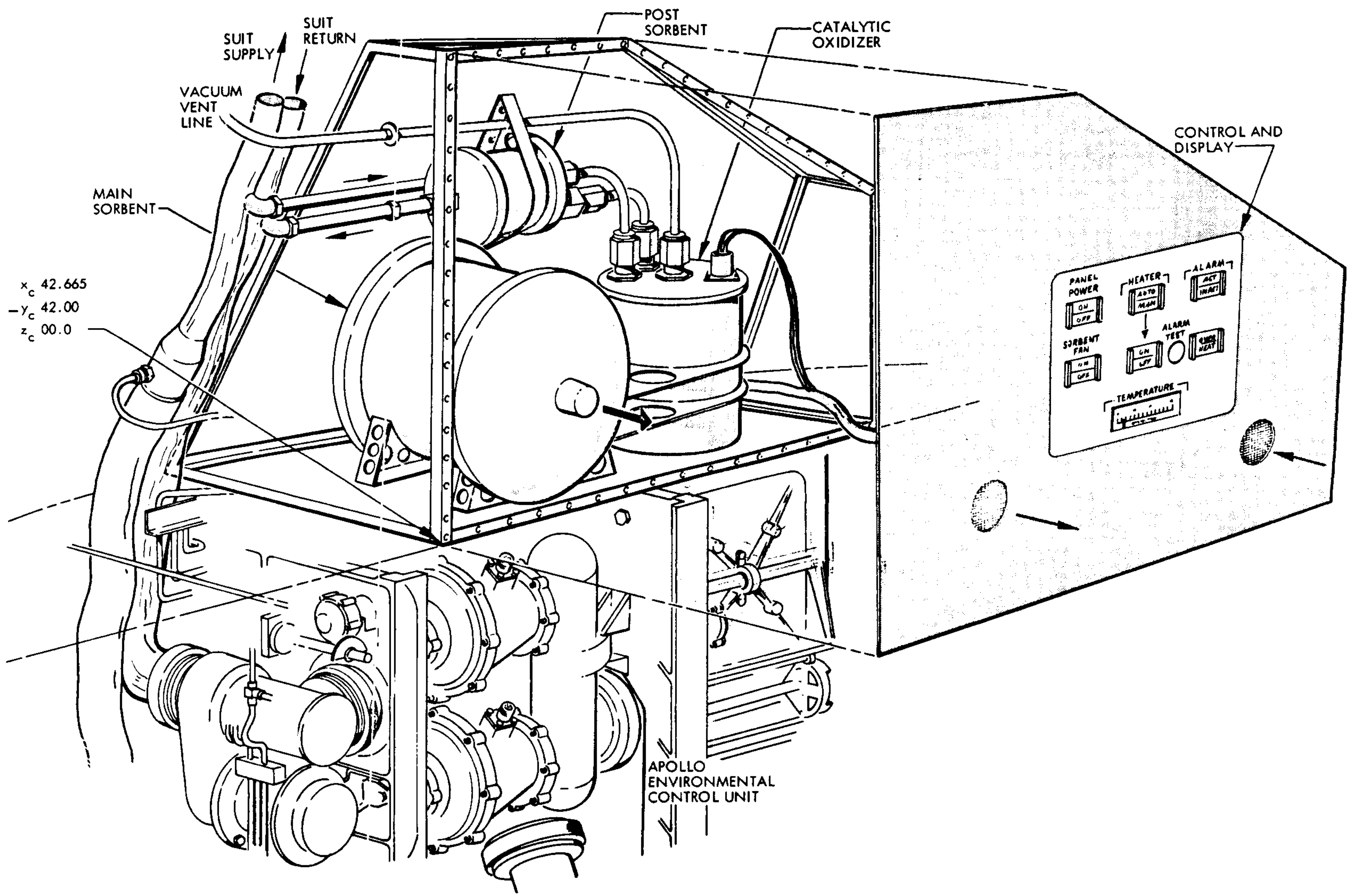

Figure 6-2 Installation Drawing, Equipment in Apollo Command Module

Figure 6-3 Mockup of Location in Apollo CM, Showing Relation of Components

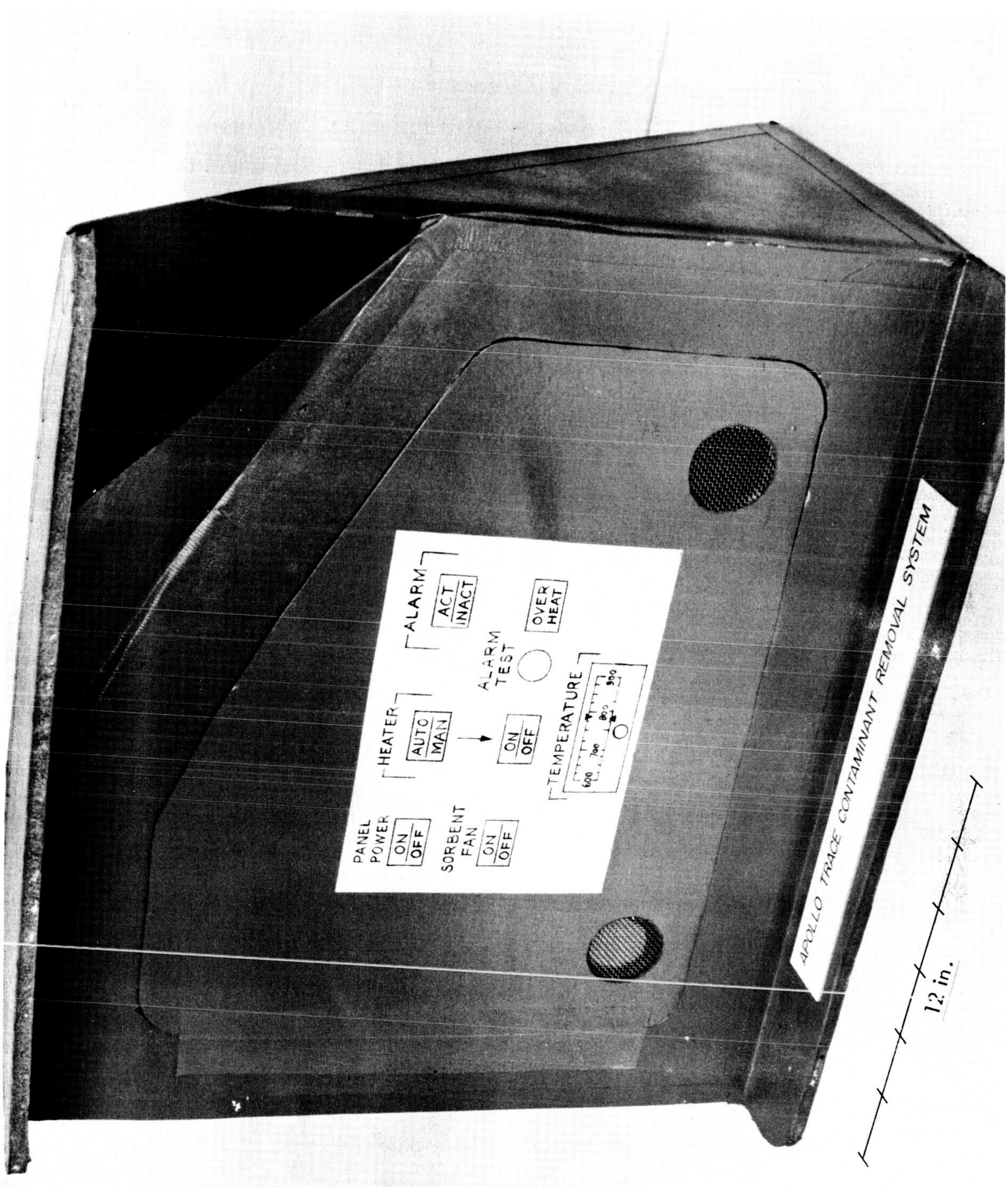

Figure 6-4: Mockup of Location in Apollo CM, Showing Controls and Displays

6.3 ADDITIONAL HARDWARE DEVELOPMENT PRIOR TO FLIGHT QUALIFICATION

The hardware developed under this program, exclusive of bracketry and items used only for test, has been designed to withstand the Apollo mission environmental conditions from launch through re-entry. Consequently, this hardware could be subjected directly to qualification testing.

During the program, however, equipment weight was not minimized in cases where significant cost savings could be made by using a non-optimum approach. In addition, the hardware was not designed specifically for a particular location in the Apollo Command Module.

During testing of the hardware at LMSC, operating and performance characteristics were established to a sufficient degree that potential improvement areas were identified.

Specific tasks which should be performed prior to entering the flight qualification phase are described below.

6.3.1 Weight Reduction

It is suggested that the following weight-reduction measures be implemented.

- Substitute bolted flanges, with titanium bolts, for the stainless steel spin-type flange rings on the main sorbent, post sorbent, and catalytic oxidizer units.
- Use lug-type flanges instead of full-diameter flanges on all units.
- Cut lightening holes in the conical end of the main sorbent canister, on the inlet end.
- Perform additional stress analyses on all components to allow thinner material gages to be used with confidence.

6.3.2 Power Reduction

The power required by the catalytic oxidizer is the result of two losses; flow-stream heating, and heat loss to ambient through internal structure and insulation. The following tasks are recommended to effect a reduction in system power drain.

- Increase the internal-to-external thermal resistance of the regenerative heat exchanger structural mounts and the insulation surrounding the heat exchanger.
- Increase the effectiveness of the regenerative heat exchanger.
- Conduct a tradeoff analysis to determine the power saving vs. increase in weight and volume implied in the above two tasks.

6.3.3 Reliability Improvements

The prototype equipment was designed for ease of disassembly to facilitate laboratory testing. In several areas of the catalytic oxidizer, spin-type closure rings and associated seals were used which are potential leakage points from the flow stream into the vacuum-type insulation. As many of these joints as possible should be permanently sealed in a flight-type unit.

A redundancy analysis should be made to determine the identity and number of parts which, for reasons of reliability, should be made redundant. A preliminary investigation indicates that the following items should probably be redundant.

- Catalytic oxidizer heater
- Catalytic oxidizer temperature control thermocouple
- Main sorbent fan

The fan and the solenoid shutoff valve supplied with the prototype hardware are qualified to MIL Specifications but not necessarily to the Apollo environmental specification. Changes, if any, in these components required to withstand the Apollo environmental conditions must be defined.

Section 7
CONCLUSIONS AND RECOMMENDATIONS

As a result of the Phase II activity the following conclusions are drawn and recommendations made.

7.1 CONCLUSIONS

Conclusions from the Phase II effort are presented below as they concern the major elements of the contaminant removal system.

7.1.1 Main Sorbent

The main sorbent canister, containing 8 lb of 4 × 10 mesh Barnebey Cheney BD charcoal impregnated at LMSC with phosphoric acid, and supplied with a flow of 10 cfm at 5 psia, was effective in removing certain contaminants as predicted in Phase I of this program.

During the 24-hr main sorbent test, with contaminants introduced at nearly 7 gm/day, it was determined that this unit had a greater dynamic removal efficiency (η_r) for contaminant removal than the average required to saturate the bed in 14 days. Since the value of η_r will decrease as the degree of saturation increases, an η_r greater than the average required should have been observed during this test.

During the five-day closed chamber test, concentrations for all contaminants primarily removed by the main sorbent were well below SMAC values. Ammonia reached a concentration greater than SMAC, indicating that the impregnated Apollo charcoal (in the LiOH canister) did not remove ammonia at the rate predicted in the Phase I report. This is attributed to the relatively high space velocity in this charcoal bed.

The phosphoric acid impregnation, responsible for ammonia removal, did not outgas sufficiently to bring this compound near SMAC during the five-day test. This impregnation appears to be a safe and effective means for ammonia removal. This type of impregnated charcoal has recently been used successfully in closed-chamber animal tests at LMSC.

During testing under "upset" conditions, contaminants removed primarily by the main sorbent canister were brought to less than SMAC in less than four hours.

During studies of potential locations in the Apollo Command Module and of interface problems with the Apollo environmental control unit, it was concluded that the most practical solution to providing 10 cfm through the main sorbent was to use a separate fan. A small, and light-weight 10 cfm fan is integral with the canister; it draws 7.5 W of power.

7.1.2 Catalytic Oxidizer – Post Sorbent

Bench tests of this unit indicated about a 20% reduction in performance of the catalytic oxidizer from the predicted performance stated in the Phase I report, with the exception of methane. Methane was oxidized with greater efficiency than was predicted, which is attributed to differences in temperature between the early laboratory and the bench tests.

The 20% reduction in performance is attributed to competition of the multiple contaminants, used in the bench test, for active catalyst sites. The earlier laboratory tests were conducted with single contaminants.

Formaldehyde and propylene were expected to be removed by the catalytic oxidizer. During part of the five-day test, when the catalytic oxidizer was inoperative, there was no abrupt increase in the concentration of these contaminants. Consequently, it appears that the catalytic oxidizer was not effective in removing them. Propylene is reasonably well adsorbed on charcoal, which accounts for it remaining at a low concentration. No explanation is apparent for formaldehyde removal by other means.

Highly toxic products of oxidation such as $COCl_2$, HCl, HF, and SO_2 were monitored during the five-day test and found to remain well below SMAC values. The Apollo LiOH canister and the post-sorbent ($LiOH/Li_2CO_3$) canister are extremely effective in removing such products of oxidation. The potential hazard of oxidation-product formation should not be considered a significant problem in the use of a catalytic oxidizer in the Apollo Command Module.

The "upset" test with CO, CH_4 and H_2 showed that CH_4 and H_2 were reduced to less than SMAC in four hours under test conditions. After four hours, CO was approximately three times SMAC in this test. Extrapolation to a zero cabin leakage condition, shown in Table 5-5, indicate that CH_4 and H_2 would probably be greater than SMAC after four hours for this case. Inability to achieve $\leq$ SMAC in four hours is due primarily to the low flow rate through the catalytic oxidizer (1.2 cfm) and secondarily to the reduced performance of this unit when exposed to multiple contaminants. It should be noted that massive quantities are required to "upset" CO, CH_4, and H_2, which are the primary contaminants removed by the catalytic oxidizer. Consequently, the "upset" performance noted in the five-day test would in all probability be sufficient for any operational upset encountered.

The catalytic oxidizer – post sorbent – shut-off valve combination can be easily accommodated in the Apollo "clothing storage area". In this location, plumbing integration with the Apollo environmental control unit (ECU) can be made with negligible modification to the present Apollo ECU.

7.1.3 Controller

The control and display unit functioned as planned and was effective as a test support item. A greatly reduced control and display panel is shown on the mockup of the Apollo Command Module location (contaminant removal equipment placed in "clothing storage area").

7.2 RECOMMENDATIONS

As a result of the activities carried out under this program the following recommendations are made.

7.2.1 Power Reduction

Due to an anticipated limitation in power available for this system, modifications to the catalytic oxidizer to reduce power consumption should be explored. This work would involve primarily an increase in the catalytic oxidizer regenerative heat exchanger effectiveness and reduction of energy losses to ambient through the case. This work would be undertaken after the Phase III catalyst studies to take full advantage of the results thereof. The catalytic oxidizer optimization analysis should be redone in the light of actual heat transfer performance data acquired during Phase II.

7.2.2 Performance Characteristics

Additional experimental work should be conducted to allow better predictions of the dynamic performance of the impregnated charcoal for contaminant removal as a function of mesh size, space velocity, and degree of saturation. This work should be conducted with multiple contaminants in the flow stream.

7.2.3 Apollo Installation

Additional consideration should be given to the installation of the developed hardware, or modifications thereof, in the Apollo Command Module so that if such an installation is required at a later date, the feasibility of so doing will be established with certainty.